

Georgia Tech Sponsored Research

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Project	E-20-F88 F767
Project director	FROST J.D. (JAMES David)
Research unit	CIVIL ENGR
Title	ACQUISITION OF INSTRUMENTATION FOR NON-CONTACT MULTI-SCALE MATERIAL RESPONSE MEASUREMENT
Project date	3/30/2004

Final Report for Period: 07/2000 - 12/2003

Submitted on: 04/15/2004

Principal Investigator: Frost, J. David .

Award ID: 0079589

Organization: GA Tech Res Corp - GIT

Title:

Acquisition of Instrumentation for Non-Contact Multi-Scale Material Response Measurement

Project Participants

Senior Personnel

Name: Frost, J. David

Worked for more than 160 Hours: Yes

Contribution to Project:

Name: Zureick, Abdul-Hamid

Worked for more than 160 Hours: Yes

Contribution to Project:

Name: Santamarina, J. Carlos

Worked for more than 160 Hours: Yes

Contribution to Project:

Name: Haj-Ali, Rami

Worked for more than 160 Hours: Yes

Contribution to Project:

Name: Kurtis, Kimberly

Worked for more than 160 Hours: Yes

Contribution to Project:

Post-doc

Graduate Student

Undergraduate Student

Technician, Programmer

Other Participant

Research Experience for Undergraduates

Organizational Partners

Other Collaborators or Contacts

Activities and Findings

Research and Education Activities:

Researchers in the Structural Engineering and Mechanics, Materials and Geosystems Engineering groups in the School of Civil and Environmental Engineering at Georgia Tech have been utilizing a range of alternative approaches as the basis for non-contact multi-scale investigations of the behavior of civil engineering materials. For example, Atomic Force Microscopy (AFM) and Optical Profile Microscopy (OPM) have been used to quantify the micro and macro surface characteristics of high-density polyethylene (HDPE) geomembranes and fiber-reinforced polymers (FRP). When conventional interface shear tests are evaluated in light of these quantitative surface measurements, differences in behavior become readily understood. Similar insights are being discovered by the writer's of this proposal using a range of techniques. While the Georgia Institute of Technology has made significant investment in equipment for conducting such measurements, there remain a range of capabilities which are urgently required by the project team to enable them to quantify critical aspects of the response of various materials and these are the focus of this proposal. In particular, resources to permit the acquisition of five systems are requested as follows: (a) an infra-red thermal imaging system; (b) an automated photoelastic stress/strain system; (c) a multi-point parallel scanning laser extensometer system; (d) a scanning white light interferometer; and (e) upgrades for an existing laser scanning confocal microscope. While these systems are all based on different operating principles, they are all capable of providing non-invasive non-contact quantitative information about material characteristics and behavior over a range of scales.

Findings: (See PDF version submitted by PI at the end of the report)

Equipment purchased to date:

DT1400 Infra-Red Thermal Imaging System (256 x 256, Liquid Nitrogen cooled)

This system consists of a very sensitive infrared camera that is combined with special high-speed digital electronics to measure small changes in a temperature field. The result is a full-field stress map of the surface. This system includes a motion compensation software and hardware; it allows for attained compensation for motions at frequencies from 0.6-400Hz. The system is also capable of following a variable amplitude (random) motion. The detector array contains thousands of on-chip integrators that collect data simultaneously, producing a near-live full-field stress image. Data collected from the infrared camera head is processed at hundreds of frames per second. The processed images can be used to measure properties that are directly related to the principal stress field and/or to temperature profile.

The DT 1400 camera head is based on an InSb focal plane array. The array is cooled to liquid nitrogen temperature. The system provides excellent DC thermal imaging, and very high quality AC performance. System includes: camera head, cables, computer, and image processing boards. Stress Photonics' DV Foundation image processing software, DeltaTherm Control Panel, and DeltaVision post-acquisition analysis data-processing software is included. Also included are tripod and carrying cases for computer and DeltaTherm.

Features of the system (in addition to those stated above):

Live, high quality DC thermal imaging with corrections for detector uniformity to make setting up microscopic and other imaging a simple matter of point, focus, and shoot. Built in oscilloscope for checking the quality of the reference signal. Thermal Resolution: 1mKelvin full-field (30s acquisition time) Constant amplitude and variable amplitude load sequences with impact triggering Loading frequencies 0.6 Hz to 400 Hz. Capable of spatial resolutions better than 23 micron per pixel. DeltaVision software for data acquisition, presentation and storage.

GFP 1200 Photoelastic Stress Analysis System

The GFP1200 Photoelastic stress/strain analysis (PSSA) System is a strain measurement system based on photoelasticity. Advanced instrumentation is used which differentiates this system from similar photoelastic techniques used historically. A specially tinted paint-on coatings is used for this purpose. A new technique is developed for measuring the thickness of a paint-on coating automatically. The same automatic polariscope that measures the strain amplitude can measure the thickness of the coating, eliminating it as an unknown. After a thin plastic coating is applied to the surface of the part, an instrument called a polariscope measures the strain induced birefringence to create a full-field strain map. A software is being developed with the system in order to allow data interpretation and post analysis.

The system includes: Enhanced Grey-Field Polariscope with automated filters. Polarized light projector. Windows NT computer with LCD monitor. Enhanced DeltaVision software for post-acquisition analysis and presentation of images. GFP virtual control panel for image acquisition. Matrox Image Acquisition Card. Tripod and tripod head. Coating Kit.

Partially helped Acquire: MTS Model 810 Material Testing System

(*) System is still in configuration mode as more peripherals are added (data acquisition, grips, wedges, etc). This system is a uniaxial hydraulic loading frame used to load structural components and material coupon samples while the GFP-1200 and Delta Therm 1400 are

monitoring the testing sample. The main features of this hydraulic loading frame are: Nominal dynamic load rating: 250 kN (55,000 lbs); 55kip Actuator; 6" Stroke Hydraulic grips with adjustable pressure; 4" and 2" wedge sets; 6-channel input and output for data acquisition; software for programming tests; Low noise 'silent flow' hydraulics; Special output card for interfacing with the Delta Therm 1400 and GFP1200 systems.

3-D Reconstruction Software

Leica 3D Reconstruction image processing software has been acquired and installed into the Leica TCS-NT LSCM existing in the School of Civil and Environmental Engineering at Georgia Tech. This instrument has been configured specifically for the three-dimensional modeling and volumetric examination of structural materials. The addition of the 3D Reconstruction software enhances the imaging capabilities of this system significantly. This software affords higher resolution than the previously existing software and offers additional options for image processing, including volumetric rendering, animations, slicing and, raytracing from confocal images. These capabilities allow for three-dimensional examination of materials at the micro- and meso-scale.

Several types of materials have been examined and modeled using the LSCM with the 3D reconstruction software, including fiber-portland cement composites, cement paste, concrete, glass-fiber reinforced polymers, and Nitinol shape-memory alloys. A video acquired from LSCM images and produced using the Leica 3D software, is being used to examine the size of air voids in concrete. Data collected from the 3-dimensional representation of glass fibers in a transparent epoxy matrix has been used to quantify fiber volume fraction, a parameter useful for predicting strength and elastic modulus of FRP samples. In addition to qualitative assessment of material properties, the Leica 3D software can be used for direct quantitative characterization of materials. One such index, the roughness number (RN), describes the roughness of a fracture surface relative to a planar surface. The data collected to date has been used to show that a correlation appears to exist between the RN measured from the fracture surface of fiber-reinforced mortars, the fiber volume fraction (0.6, 1.2, and 2.0%), and the flexural toughness of the cement-based composite.

Motorized X-Y Stage

An indexable stage, with a customized sample holder, has been designed and procured through Vashaw Scientific. The sample holder has been customized to (1) allow confocal images to be acquired in both reflected and transmitted light and (2) allow a unique orientation of samples on a cylindrical base, providing for precise repositioning. Delivery of the motorized stage is expected in May 2001.

The stage has been configured to allow accurate repositioning of samples (to 1 μ m), permitting samples to be examined, removed, replaced precisely, and re-examined over extended time periods. Sample positions are read through a digital readout and can be recorded for accurate repositioning. This stage will facilitate precise examination of damage process zones in materials in response to chemical and physical attack, which can be induced on the stage using the climate/load chamber and ex situ loading or environmental exposure, which will require that the sample be removed, tested, and accurately repositioned for examination using an existing LSCM.

Training and Development:

Project participants have gained valuable insight into the operation and performance of various competing measurement systems. The instruments have been used by both undergraduates and graduate students for research. Some of the students impacted are listed below.

Undergraduates:

Mr. Shane Johnson, Fall 2003 - present
 Mr. Anthony Fisher, Fall 2002 - present, African American
 Ms. Trenise Trent, Fall 2003, African American
 Mr. Jason Ideker, Summer 2001 - Spring 2002
 Ms. Gayle Willis, Spring 2001 - Spring 2002

Graduate students:

Mr. Ahmet Citipitiuglo
 Ms. Courtney Lloyd Collins
 Mr. Rani El-Hajjar
 Dr. Hakan Kilic
 Ms. Ji-Yeon Kim
 Mr. Ben Mohr
 Ms. Hanifah Anastasia Muliana
 Mr. Mauricio Lopez
 Ms. Nikhila Naik
 Ms. Kristin Thibodeaux

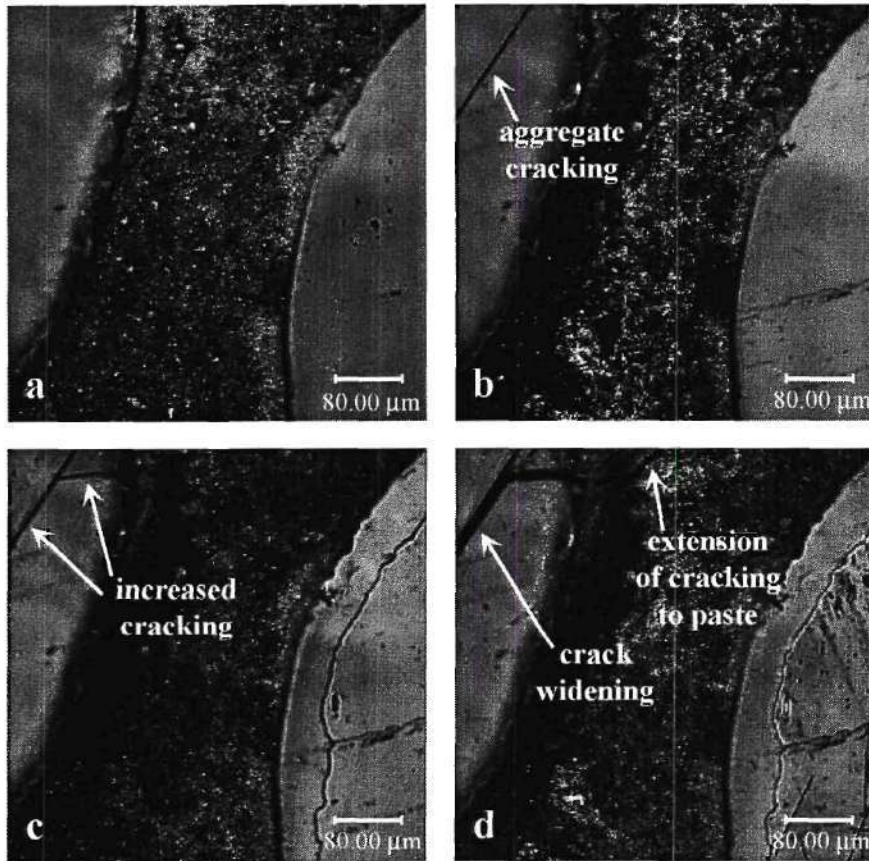


Figure 1: LSCM images of crack formation and progression in the reference sample at (a) 2, (b) 7, (c) 14, and (d) 26 days

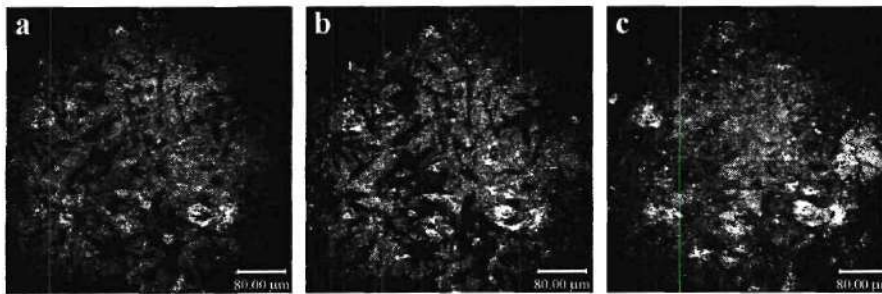


Figure 2: LSCM images of the paste/aggregate interface in samples prepared with LiNO_3 , $[\text{Li}_2\text{O}]/[\text{Na}_2\text{O}_{\text{eq}}]=0.5$ at (a) 3, (b) 8, and (c) 14 days

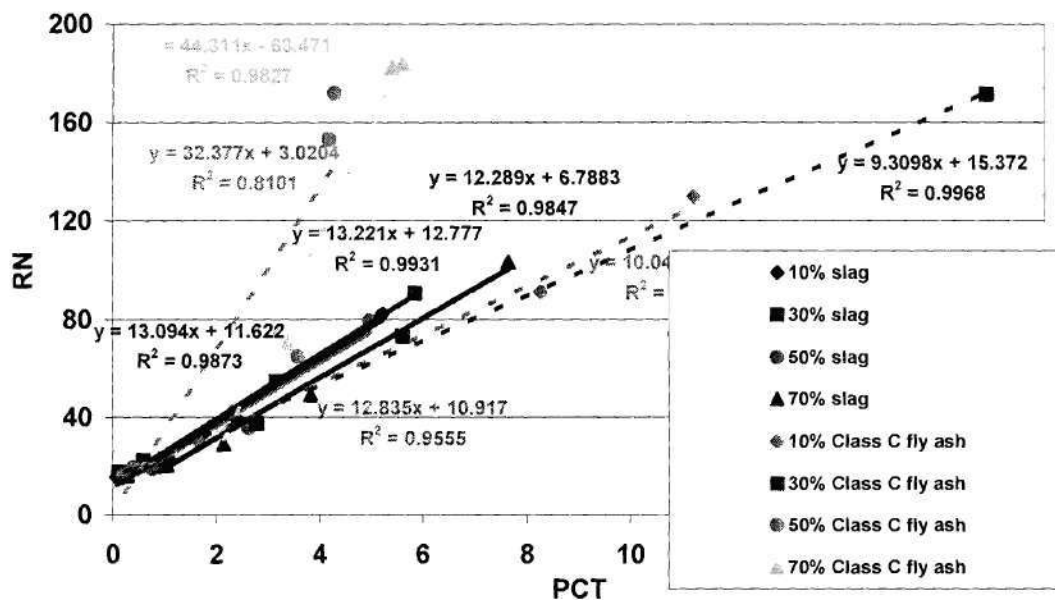


Figure 3: Roughness number vs. post-cracking toughness for fiber-cement fracture surfaces; samples contain varying amounts of slag and Class C fly ash.

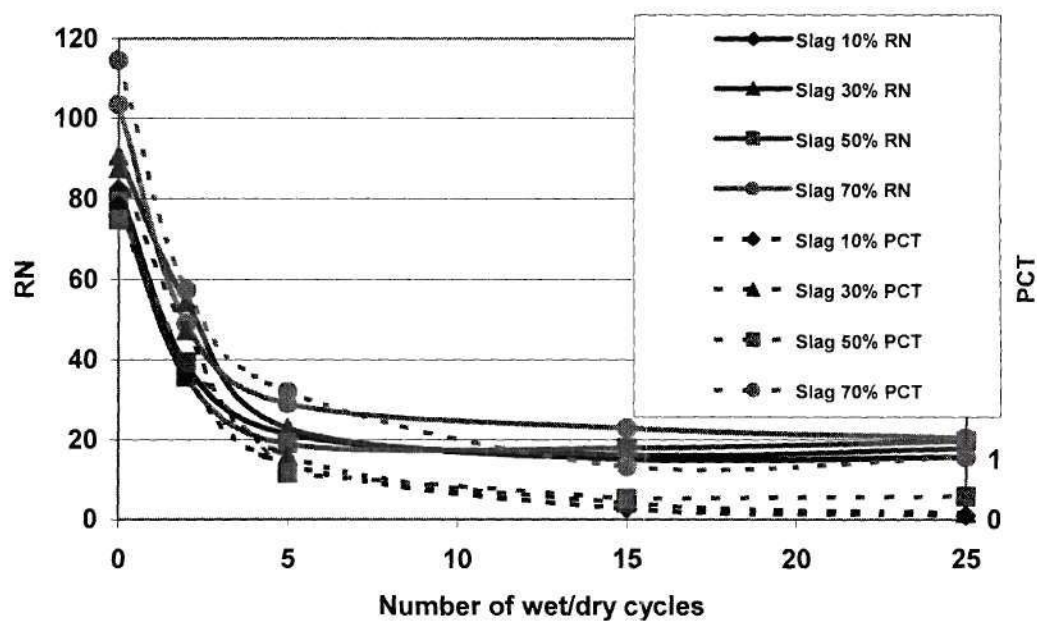


Figure 4: Roughness number and post-cracking toughness measured in fiber-cement samples of the same age decreases with increasing number of wet/dry cycles.

Outreach Activities:

A number of technical presentations have resulted from this MRI grant, in addition to journal publication. These presentations have been given in a range of forums, increasing awareness of our ongoing collaborative activities in non-contact materials characterization research. These presentations include:

Conference Presentations

El-Hajjar, R. F., and Haj-Ali, R. M., (2003), 'Surface Strain Measurement in Pultruded Composites using Infra Red Thermography', Tenth International Conference on Composites/Nano Engineering ICCE-10, New Orleans, LA, July 20-26

El-Hajjar, R. F., and Haj-Ali, R. M., (2003), 'Fracture Toughness and Crack Growth in FRP Pultruded Composites,' 16th ASCE Engineering Mechanics Conference, 16th ASCE Engineering Mechanics Conference, University of Washington, Seattle, WA, July 16-18

Kilic, H. and Haj-Ali, R., 'Nonlinear Elastic-Degrading Models for the Analysis of Pultruded Composites', SAMPE 2003 (48th ISSE), May 11-15, 2003, Long Beach, CA, pp. 1036-1048.

El-Hajjar, R. F., and Haj-Ali, R. M., (2003), 'A Quantitative Thermoelastic Stress Analysis Method for Pultruded Composites,' 16th ASCE Engineering Mechanics Conference, University of Washington, Seattle, WA, July 16-18

El-Hajjar, R.F. and Haj-Ali, R. M., (2003), 'Infrared (IR) Thermography for Strain Analysis in Pultruded Fiber Reinforced Plastics,' Society for Experimental Mechanics Annual Conference, Charlotte, NC, June 2-4

Citipitioglu, A. M., El-Hajjar, R. F., and Haj-Ali, R. M., (2002), 'Detailed 3D Simulation of Bolted Pultruded Composite Connections,' 15th ASCE Engineering Mechanics Conference, Ed., Smyth A., Columbia University, New York, NY, June 2-5

El-Hajjar, R. F., and Haj-Ali, R. M., (2002), 'Fracture Testing and Micromechanical Analysis of Pultruded Composites,' 15th ASCE Engineering Mechanics Conference, Ed., Smyth A., Columbia University, New York, NY, June 2-5

Muliana, A. H., and Haj-Ali, R. M., 'Nonlinear Viscoelastic Micromechanical Models for the Analysis of Pultruded Composites,' Proceedings of Tenth International Conference on Composites/Nano Engineering (ICCE-10), July 20-26, 2003

Muliana, A. H., and Haj-Ali, R. M., 'A Micromechanical Model for the Nonlinear Viscoelastic Behavior of Laminated Composites,' Proceedings of the 16th ASCE Engineering Mechanics Conference (EM2003), Seattle, Washington, July 2003

Muliana, A. H., and Haj-Ali, R. M., 'Three Dimensional Micromechanical Framework for the Nonlinear Viscoelastic Behavior of Pultruded Composite Materials,' CD Proceedings of the 15th ASCE Engineering Mechanics Conference (EM2002), Ed., Smyth A., Columbia University, New York, NY, June 2-5, 6 pages, 2002

Haj-Ali, R., Kilic, H., 'A Nonlinear Constitutive Framework for Pultruded Composites', Ninth International Conference on Composites Engineering ICCE/9, ed. D. Hui, July 1-7, 2002, San Diego, CA.

K.E. Kurtis, 'Microscopy of Cement-based Materials: Should We Consider a Biological Approach?', Advances in Cement and Concrete IX: Volume Changes, Cracking, and Durability, Copper Mountain, Colorado, Aug. 10-14, 2003.

K.E. Kurtis 'New Probes Applied to Cement-based Materials', 14th ACBM/NIST Computer Modeling Workshop, Gaithersburg, June 12, 2003.

N.H. El-Ashkar (speaker) and K.E. Kurtis, 'Modified Wood Pulp Microfibers in Cement-Based Composites', 4th Annual Composites Education and Research Center (CERC) Technical Review, November 8, 2001.

Technical Presentations

K.E. Kurtis, 'Overview of Ongoing Cement-based Materials Research at Georgia Tech: Microstructure and Durability', Halliburton, Duncan, OK, October 22, 2002.

K.E. Kurtis, 'Overview of Ongoing Cement-based Materials Research at Georgia Tech: Microstructure and Durability', Chemical Engineering

Seminar, Tennessee Tech, Cookeville, TN, July 26, 2002.

K.E. Kurtis, 'Overview of Ongoing Cement-based Materials Research at Georgia Tech', W.R. Grace, Boston, MA, May 20, 2002.

'Fracture Behavior of FRP Pultruded Composites,' ASTM Committee E08 on Fatigue and Fracture, Kansas City, MO, May 2003

'Mode-I Fracture in Pultruded Composites,' Fourteenth U.S. National Congress of Theoretical and Applied Mechanics, Blacksburg, Virginia, June 2002

'A Thermoelastic Stress Analysis Method for Thick-Section Composites', Structural Engineering and Mechanics Seminar Series, School of Civil and Environmental Engineering, Georgia Institute of Technology, November 2002

'Crack Propagation Analysis for Pultruded Materials', Mechanics of Materials Seminar Series, Georgia Institute of Technology, February 2002

Journal Publications

K.E. Kurtis, N.H. El-Ashkar, C.L. Collins, and N.N. Naik, "Examining Cement-Based Materials by Laser Scanning Confocal Microscopy", Cement & Concrete Composites, p. 695, vol. 25, (2003). Published

C.L. Collins, J.H. Ideker, and K.E. Kurtis, "Laser Scanning Confocal Microscopy for In Situ Monitoring of Alkali-Silica Reaction", Journal of Microscopy, p. 149, vol. 213, (2004). Published

C.L. Collins, J.H. Ideker, G.S. Willis, and K.E. Kurtis, "Examination of the Effects of LiOH, LiCl, and LiNO₃ on Alkali-Silica Reaction", Cement and Concrete Research, p. , vol. , (). Accepted

El-Hajjar, R. F., and Haj-Ali, R. M., "A Quantitative Thermoelastic Stress Analysis Method for Pultruded Composites", Composites Science and Technology, p. 967, vol. 63, (2003). Published

El-Hajjar, R. F., and Haj-Ali, R. M., "Infrared (IR) Thermography for Strain Analysis in Pultruded Fiber Reinforced Plastics", Experimental Techniques, 2003 Society for Experimental Mechanics (SEM) International Student Paper Competition, 2nd Place entry, p. , vol. , (). Accepted

El-Hajjar, R. F. and Haj-Ali, R. M., "Mode-I Fracture Toughness Testing of Thick Section FRP Composites using the ESE(T) Specimen", Engineering Fracture Mechanics, p. , vol. , (). Submitted

Haj-Ali, R. M., Muliana, A. H., "Numerical Finite Element Formulation of the Schapery Nonlinear Viscoelastic Material Model", Int. J. of Numerical Method in Engineering, p. 1, vol. 59, (2004). Published

Haj-Ali, R. M. and El-Hajjar, R. F., "Crack Propagation Analysis of Mode-I Fracture in Pultruded Composites using Micromechanical Constitutive Models", Mechanics of Materials International Journal, p. 885, vol. 35, (2003). Published

Books or Other One-time Publications

K.E. Kurtis, "Microscopy of Cement-based Materials: Should We Consider a Biological Approach?", (2003). Book, Published
 Editor(s): D. Lange, K. Scrivener, J. Marchand
 Collection: Advances in Cement and Concrete IX: Volume Changes, Cracking, and Durability
 Bibliography: Copper Mountain, Colorado, Aug. 10-14

El-Hajjar, R. F., and Haj-Ali, R. M., "Surface Strain Measurement in Pultruded Composites using Infra Red Thermography", (2003). conference proceedings, Published
 Collection: Tenth International Conference on Composites/Nano Engineering ICCE-10
 Bibliography: New Orleans, LA, July 20-26

El-Hajjar, R.F. and Haj-Ali, R. M., "Infrared (IR) Thermography for Strain Analysis in Pultruded Fiber Reinforced Plastics", (2003).

conference proceedings, Published
Collection: Society for Experimental Mechanics Annual Conference
Bibliography: Charlotte, NC, June 2-4

Web/Internet Site

Other Specific Products

Contributions

Contributions within Discipline:

- The use of 'through aggregate' imaging by laser scanning confocal microscopy (LSCM), a technique for imaging reactions in concrete through glass aggregate, was developed and was shown to be effective for examining alkali-silica reaction in situ. Three-dimensional representations of the aggregate, images of reaction product both within cracks and at the paste/aggregate interface, and quantitative measurement of gel ring thickness at the surface are all examples of types of information gained by LSCM that have not, at this point in time, been possible with other microscopy methods.
- An experimental and analytical study was carried out to characterize the fracture behavior of fiber reinforced plastic (FRP) pultruded composites. A three-dimensional (3D) micromechanical constitutive model was developed and calibrated for the composite material system. This nonlinear constitutive model was a combination of nested micromechanical models for the roving and CFM layers. The ability of the proposed micromodel to predict the effective elastic properties as well as the nonlinear response under multi-axial stress states was verified and compared to the stress-strain response from off-axis tests.

Contributions to Other Disciplines:

Contributions to Human Resource Development:

Many graduate and undergraduate students have utilized the equipment acquired under this MRI award. This equipment has very much facilitated their development as researching engineers. The names of the students who have benefitted from this equipment are listed elsewhere in this report.

Contributions to Resources for Research and Education:

Contributions are described elsewhere

Contributions Beyond Science and Engineering:

Categories for which nothing is reported:

Organizational Partners

Any Web/Internet Site

Any Product

Contributions: To Any Other Disciplines

Contributions: To Any Beyond Science and Engineering

Session 4:

New Microstructure Characterization Techniques

Microscopy of cement-based materials: Should we consider a biological approach?

Kim Kurtis, Georgia Tech

The X-ray microscope:

A new tool for determining chloride ion diffusion in hardened concrete

Tom Van Dam, Michigan Tech

Imaging hydraulic cement microstructure by scanning electron microscopy

Paul Stutzman, NIST

Computed tomography from x-ray images taken along an arbitrary path

Chris Debrunner, Colorado School of Mines

ECI Advances in Cement and Concrete IX

Copper Mountain, Colorado

August 12, 2003

8:45-12:00

Some similar challenges with characterization

Both biological materials and cement-based materials characteristically:

- are in part or are wholly optically opaque
- are complex materials that are heterogeneous on multiple scales (nano → micro → meso → macro)
- contain fine features
- exist in a hydrated state
- can change (sometimes rather rapidly) with time

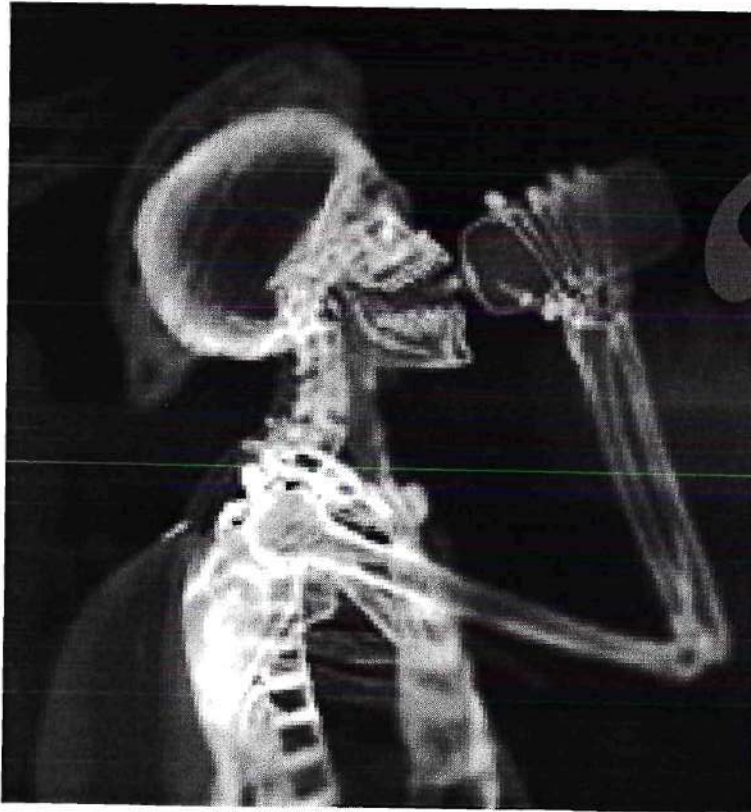
Microscopy of cement-based materials: Should we consider a *biological* approach?

Kim Kurtis

School of Civil and Environmental Engineering
Georgia Institute of Technology
Atlanta, Georgia

ECI Advances in Cement and Concrete IX
Copper Mountain, Colorado
August 12, 2003

Why would we even consider treating cement-based materials like *biological* materials?

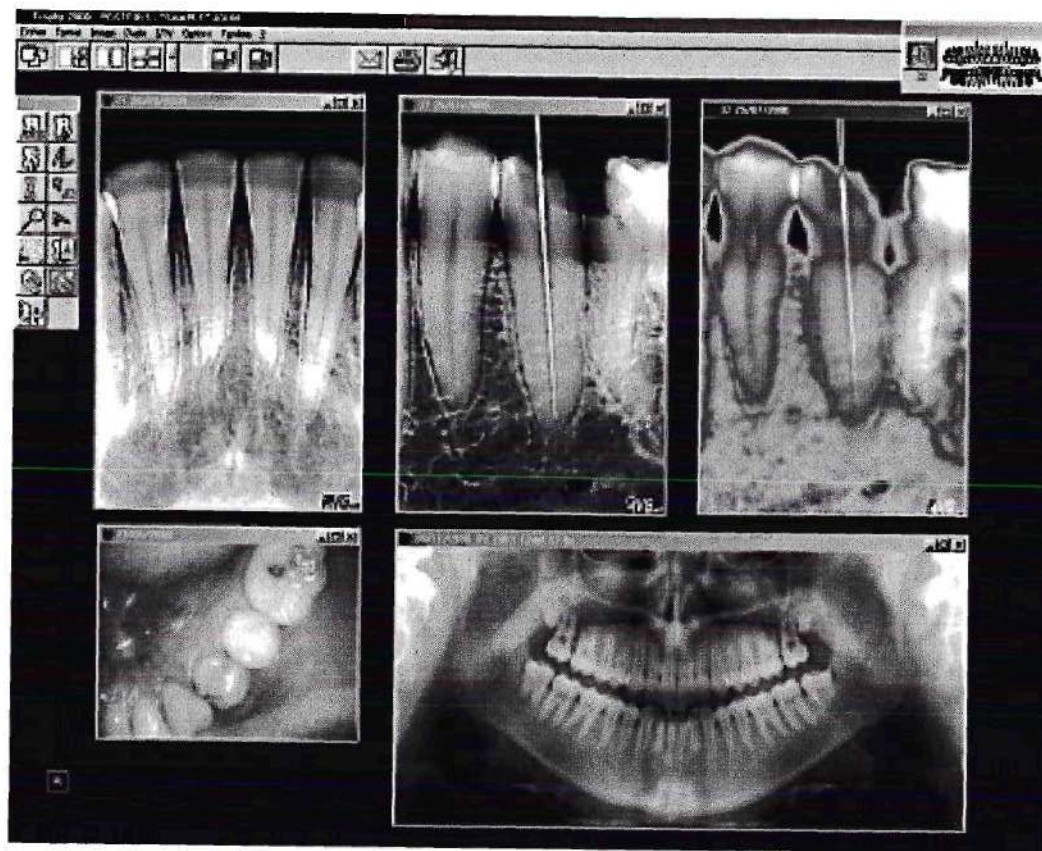


<http://www.nature.com/nsu/011011/011011-18.html>

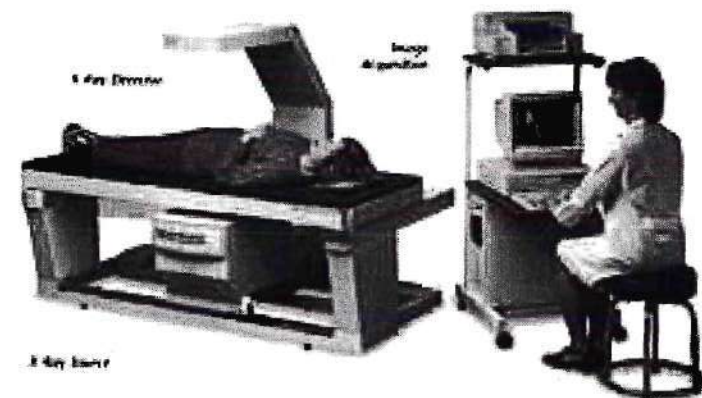
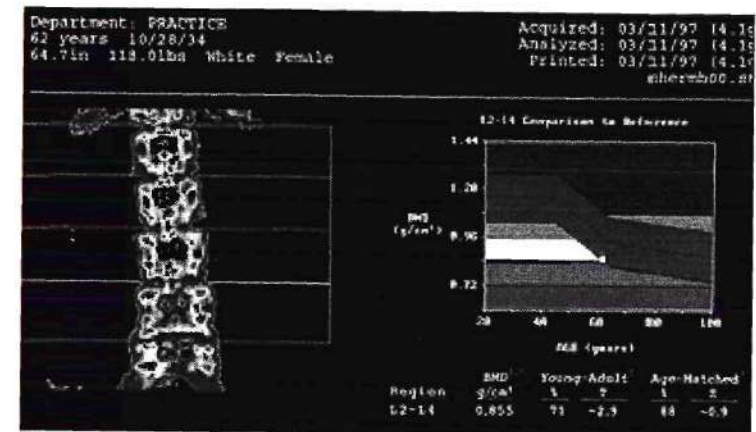


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Why would we even consider treating cement-based materials like *biological* materials?



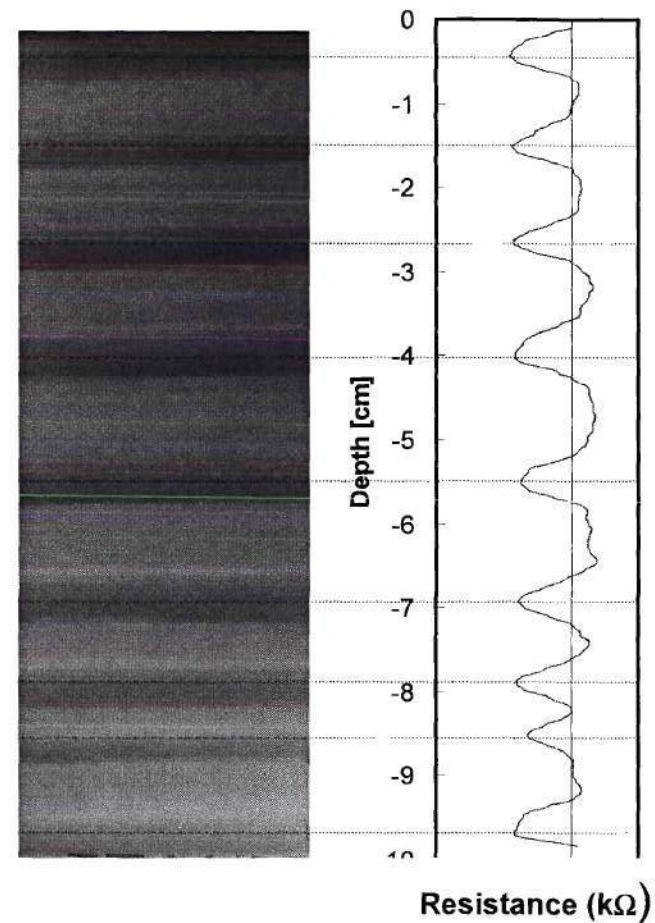
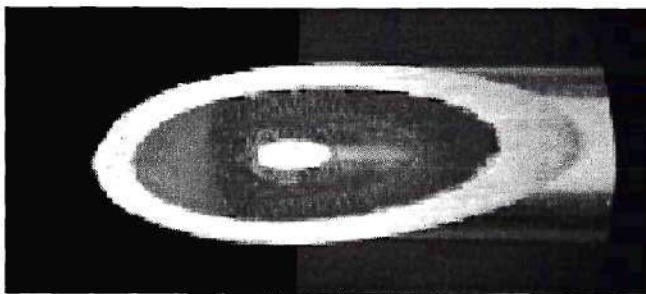
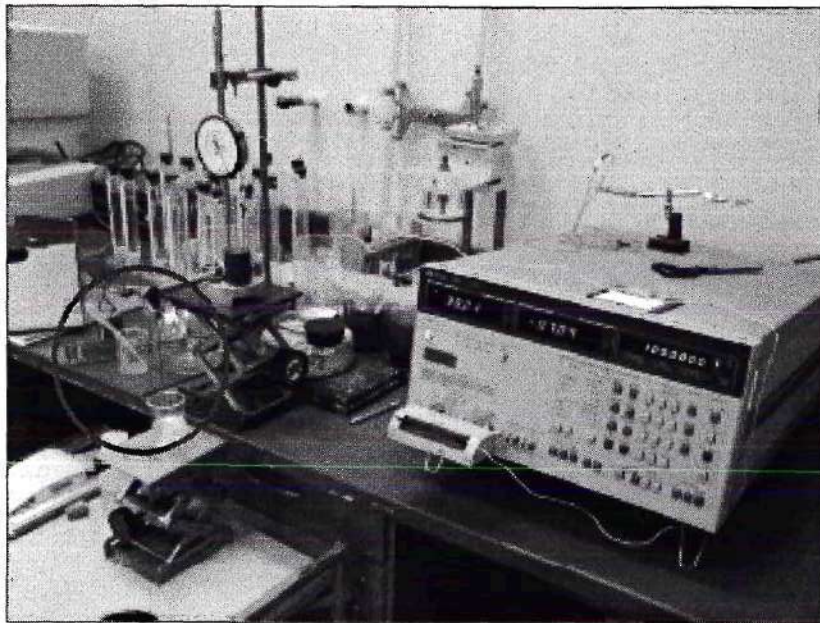
http://www.practiceworks.com/Dental_pages/Trophy_rvg.asp



imagingis.com/graphics/osteoporosis/osteoporosis1.gif

One cannot conceive anything so strange and so implausible that it has not already been said by one “materials researcher” or another.

- Descartes



C. Santamarina and co-workers, Georgia Tech

Why would we even consider treating cement-based materials like *biological* materials?

- (1) Some similar inherent difficulties must be overcome in characterizing both classes of materials.
- (2) Resource allocation is certainly much greater, and hence progress more rapid, for development of imaging techniques in medicine and the life sciences than in cement-based materials.

Some similar challenges with characterization

An “ideal” imaging method would:

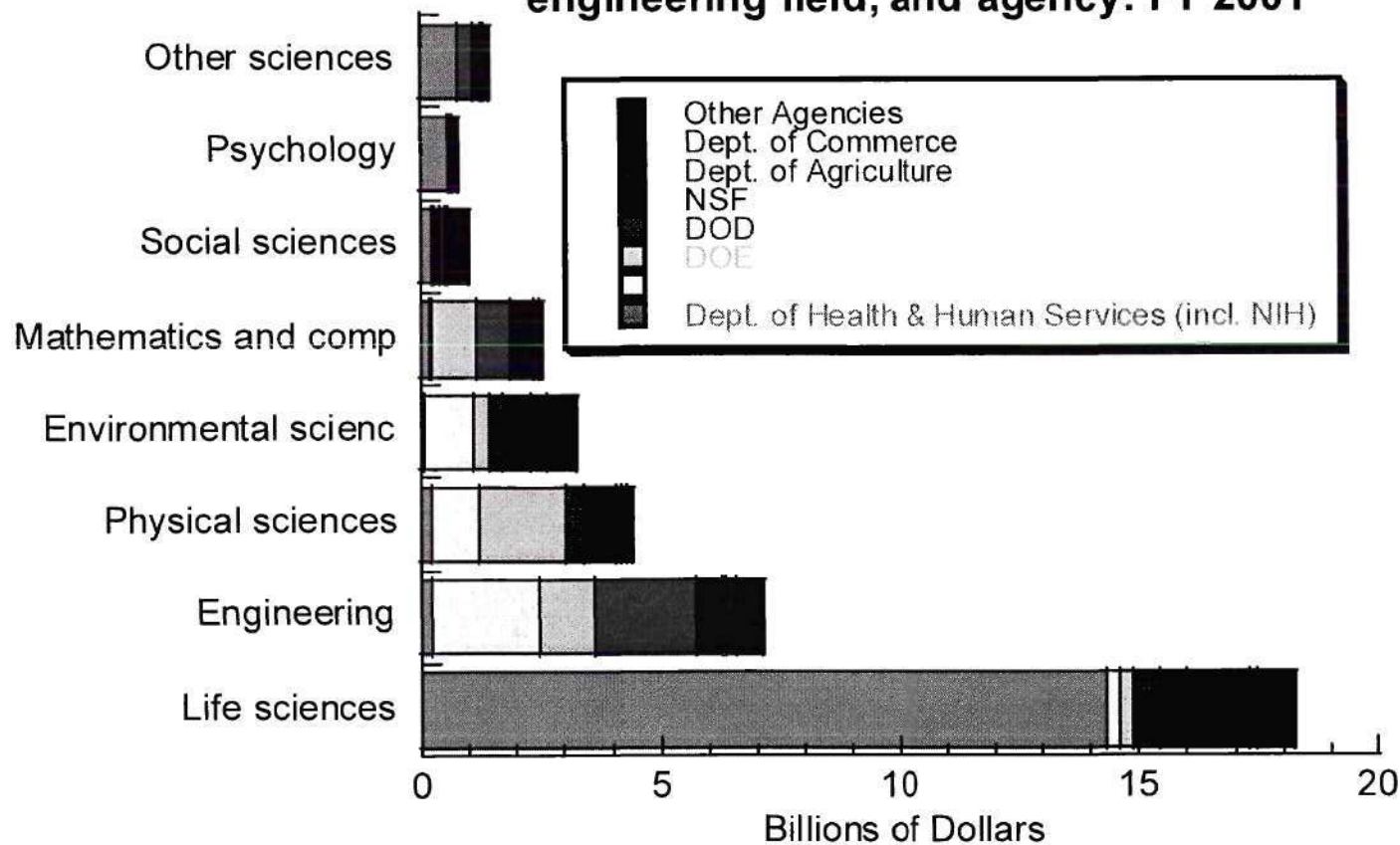
- allow sub-surface or volumetric characterization
- be able to resolve the smallest features of the structure, while still capturing the different spatial structures that comprise the whole
- allow characterization without destroying or drying the sample
- allow rapid image acquisition

In order to meet these challenges, advances in technology will often be required

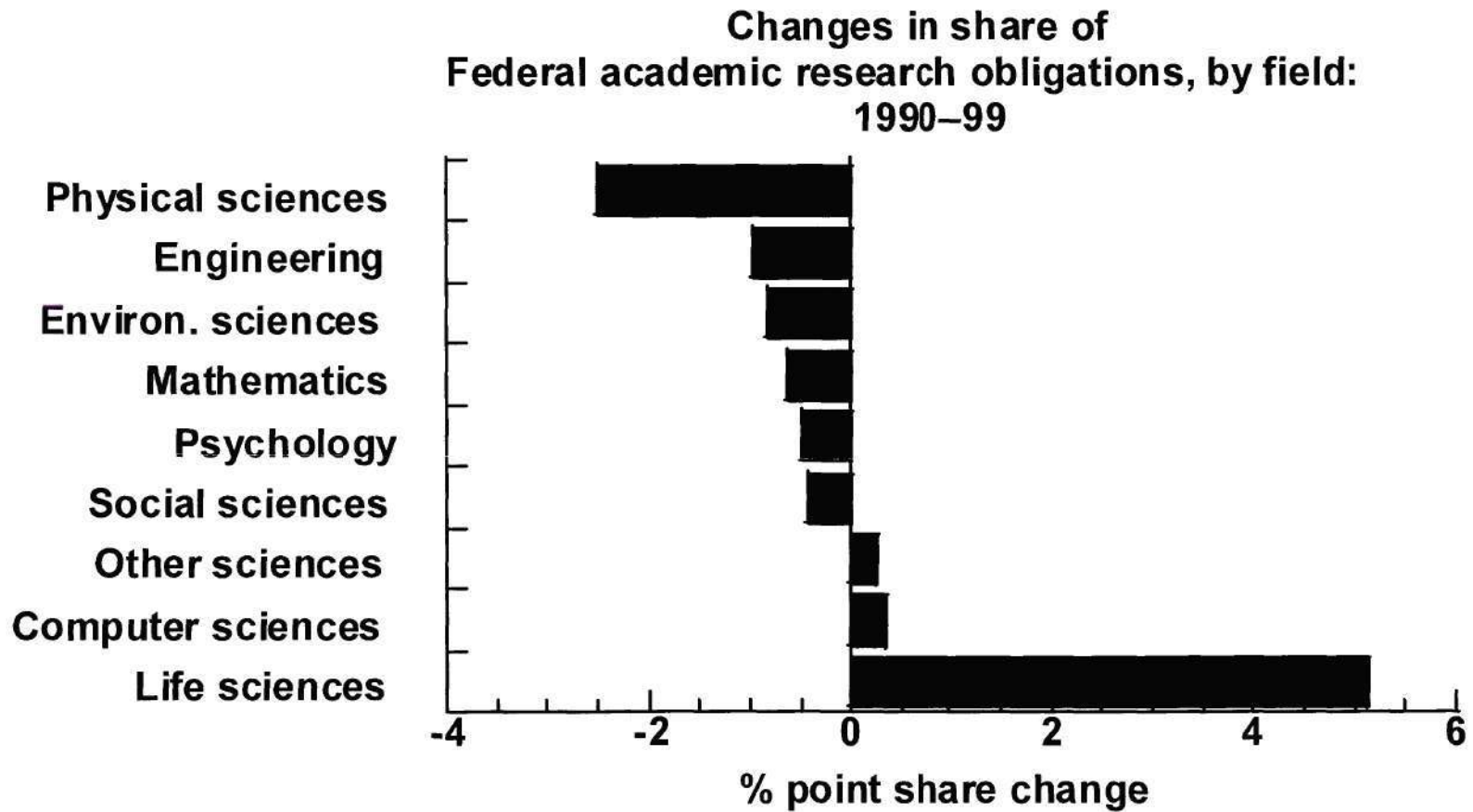
Resource allocation

While the construction industry and the health care industry both contributed ~5% to the U.S. GDP over the period 1987-2001, recent federal funding for research in the life sciences is nearly *three times* that in *all of engineering*.

Federal obligations for research, by major science and engineering field, and agency: FY 2001



Trends in resource allocation



New imaging technologies

This economic data leads to the likely conclusion that breakthrough imaging technologies will be developed for applications in the biological sciences, rather than for the study of engineering materials.

It is likely to be advantageous, then, to look at existing and emerging

- sample preparation techniques
- characterization methods
- and image analysis methods

in the life sciences and medicine to assess if they may allow for some enhancement in study of the structure and performance of cement-based materials.

Characterization Methods

Microtomography (μ CT)

Transmission Soft X-ray microscopy (TXRM)

Laser scanning confocal microscopy (LSCM)

Discussion of each technique will include:

- Principles of operation
- Typical uses
- Advantages and limitations
- Applications to cement-based materials

Development of novel uses of such techniques should be viewed as complements to existing microscopy methods, rather than as replacements.

Techniques

Microtomography (μ CT)

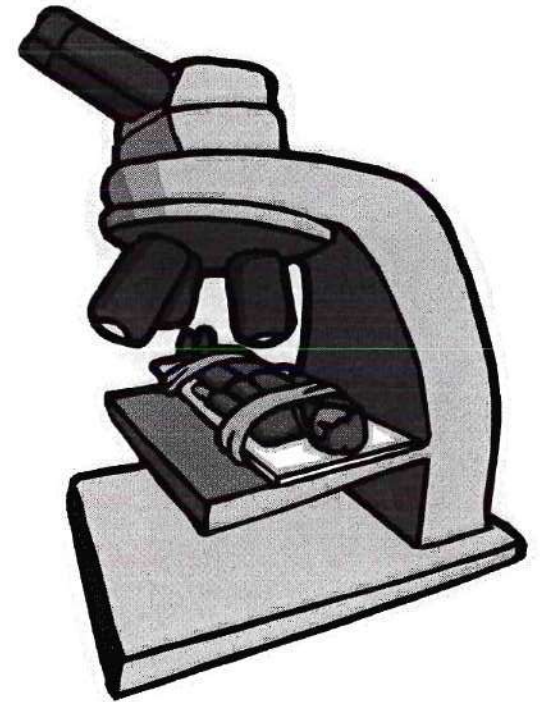
Collaborators:

PIs: Kimberly Kurtis, Stuart Stock,
Angus Wilkinson

Post Doc: Andrew Jupe

PhD Student: Nikhila Naik

REU Students: Courtney Dornell,
Luke Kennison



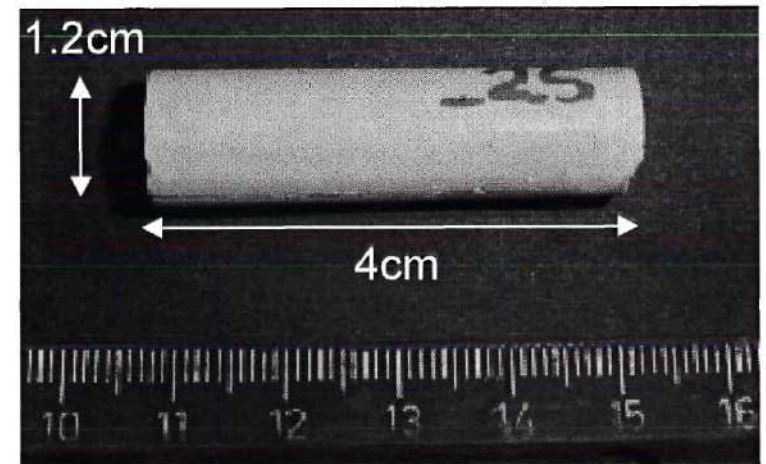
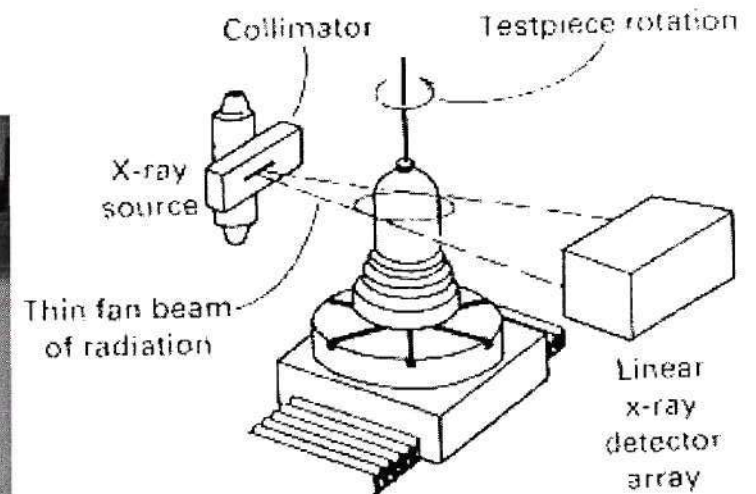
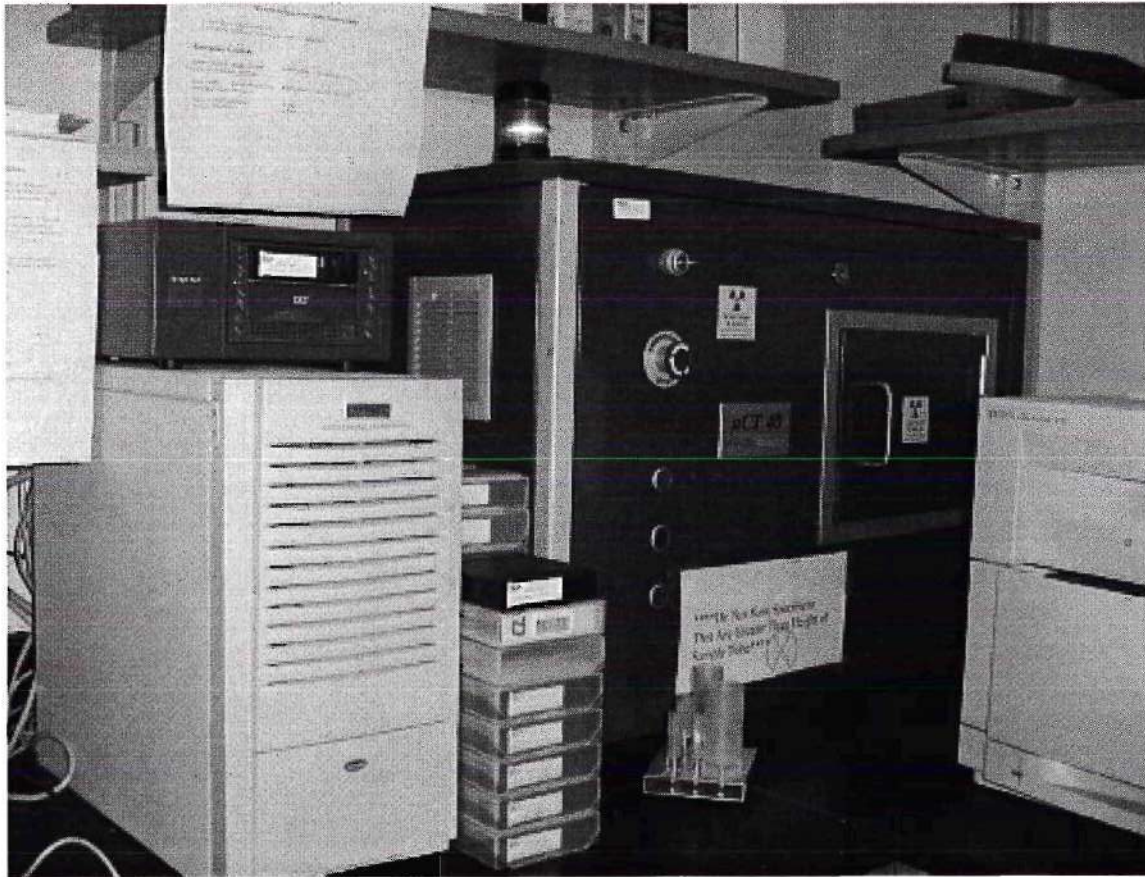
This material is based upon work supported by the National Science Foundation under Award CMS-008482. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the NSF.

Tomography

- 1979 Nobel prize for physiology and medicine was awarded to Allan Cormack and Godfrey Hounsfield for their independent work leading to the development of the first CAT scanning device.
- Today, x-ray computed tomography (also known as CAT or CT scanning) is a familiar medical procedure.
- While most modern medical CT scanners have spatial resolutions on the order of millimeters, manufacturers of desktop microtomography scanners claim resolutions to several micrometers.

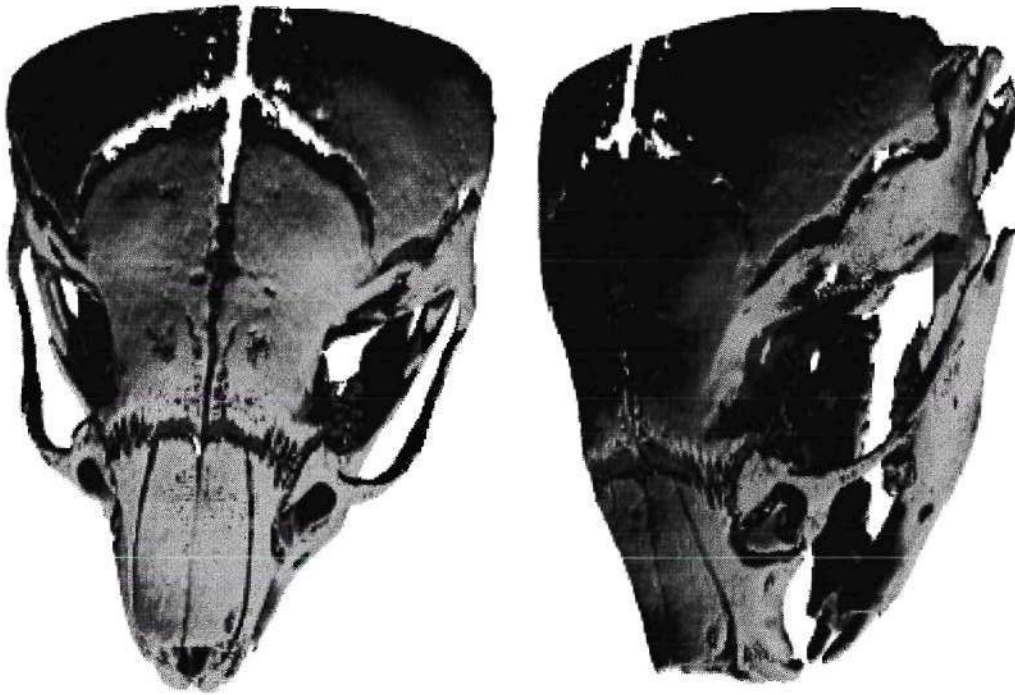
Microtomography

SCANCO MicroCT system



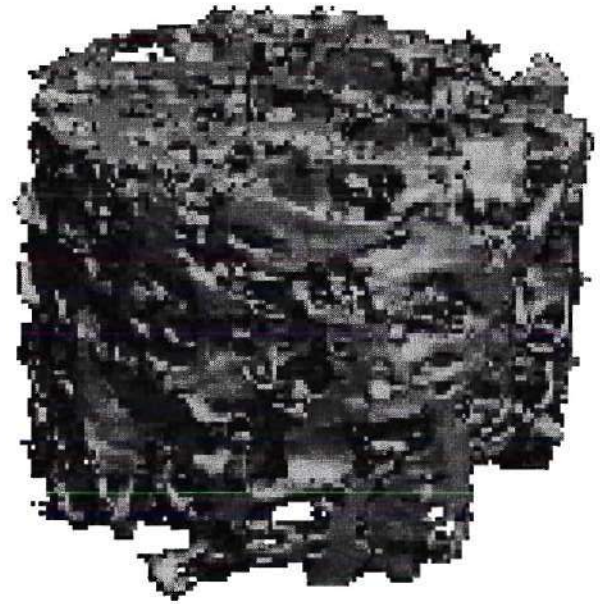
Equipment purchased under National Science Foundation grant OIA-9977551

Microtomography



MicroCT image of a mouse skull

S.R. Stock, P.H. Stern, Northwestern University



Trabecular bone scaffold

R. Goldberg and co-workers, Georgia Tech

Microtomography

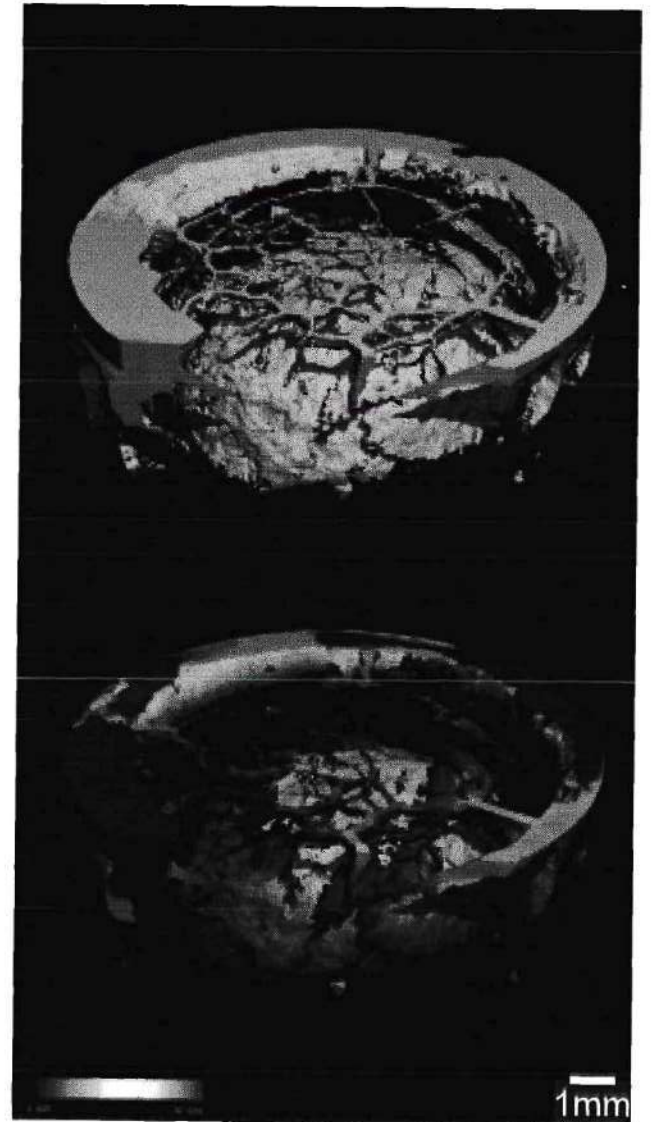
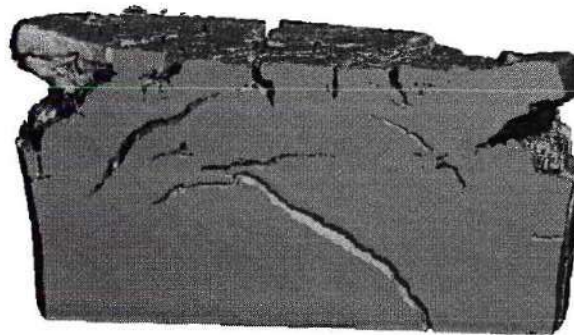
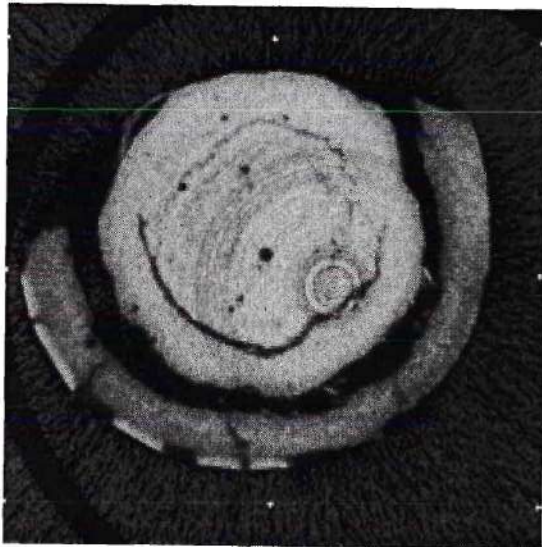
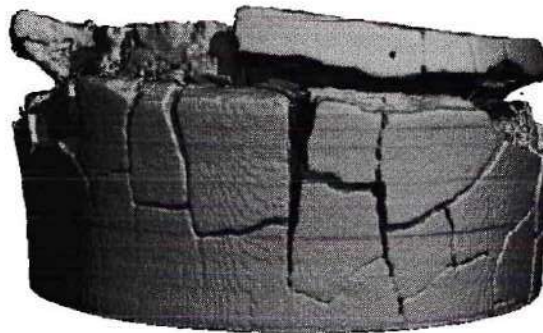
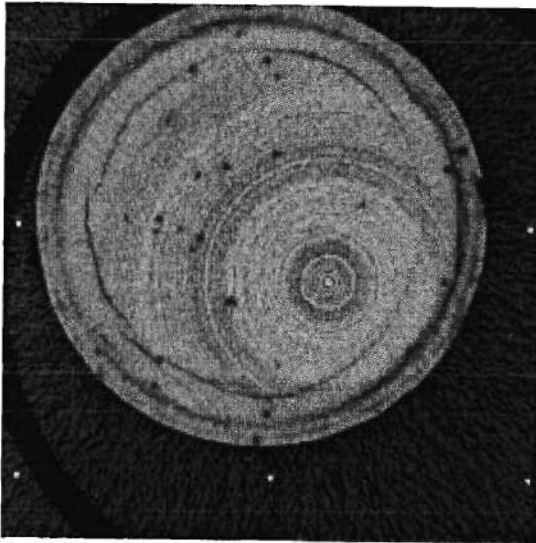
Advantages

- Lab-based x-ray examination of small, optically opaque materials
- Non-destructive
- Efficient collection of data
- Good resolution ($<10\mu\text{m}$)
- Generate digital, volumetric representation of sample
- Observe evolution of physical manifestations of damage over time

Limitations

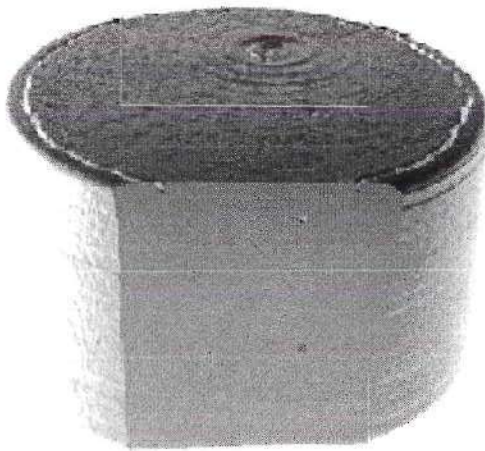
- Relatively small samples
- Data processing can be time consuming
- Some operator input during processing

Microtomography

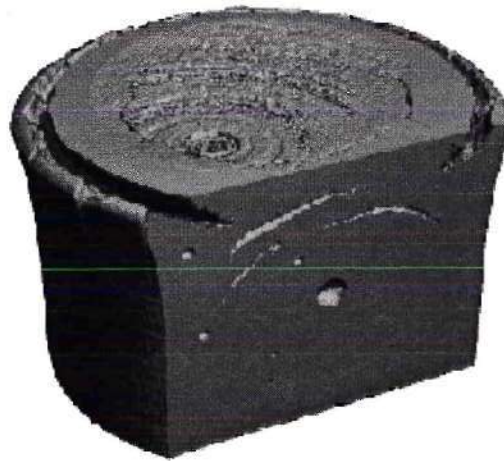


Microtomography

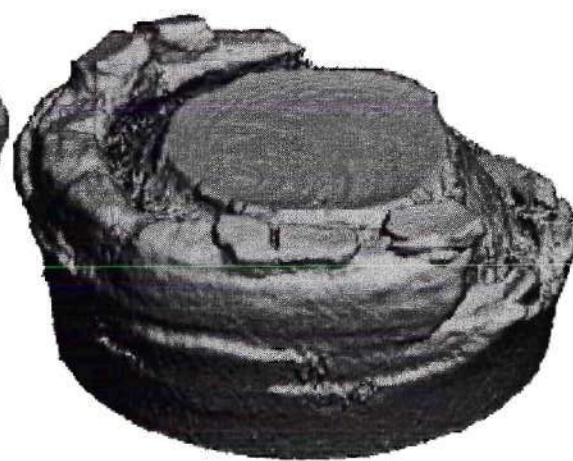
10,000 ppm sulfate
 Na_2SO_4 solution
Type I cement



w/c 0.45
Damage 1



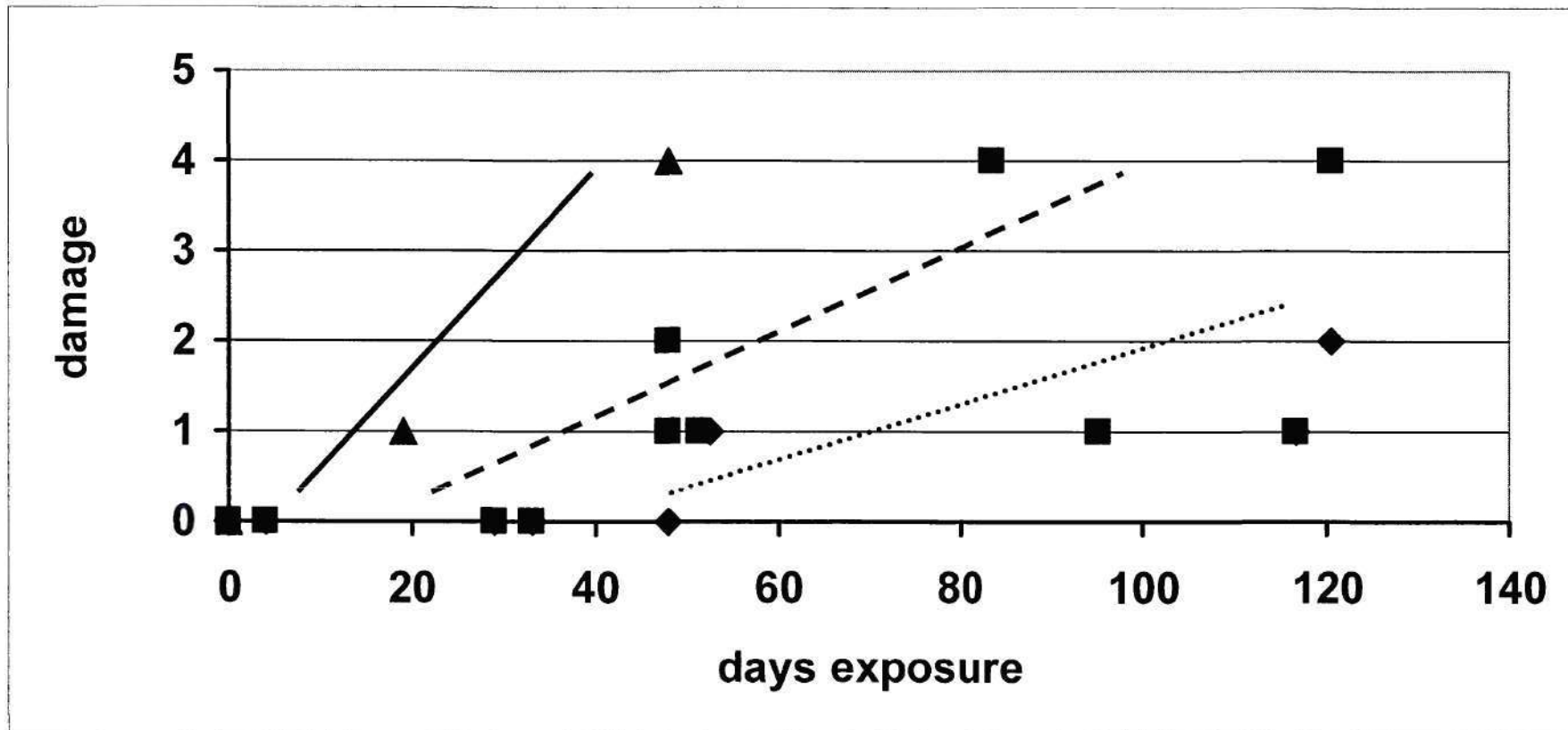
0.50
2



0.60
3

S.R. Stock, N.N. Naik, A.P. Wilkinson, and K.E. Kurtis, *Cement and Concrete Research*, October 2002, V.32:1673-5.

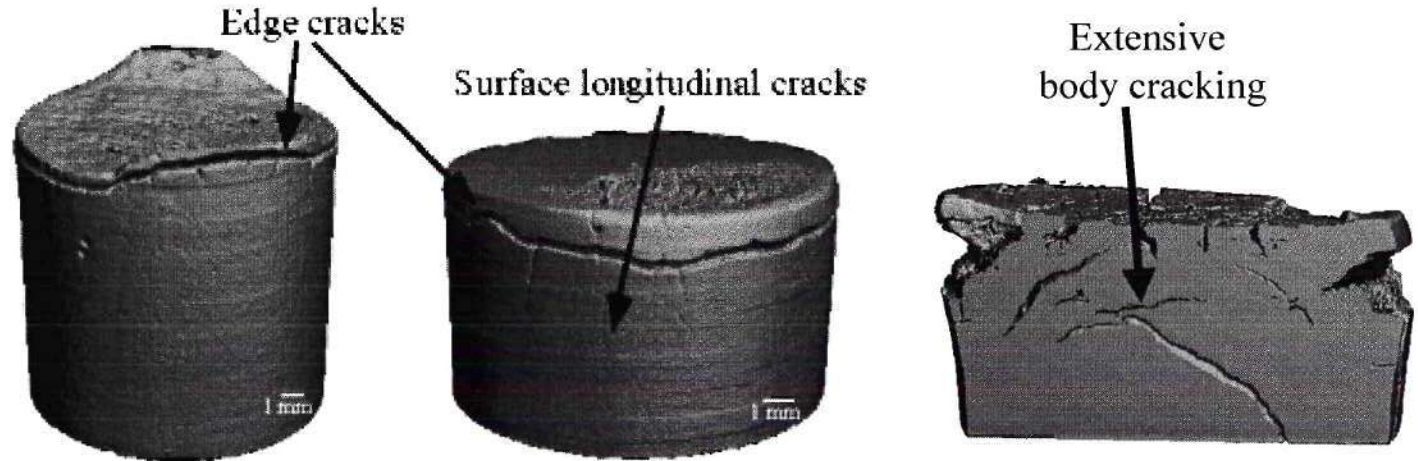
Microtomography



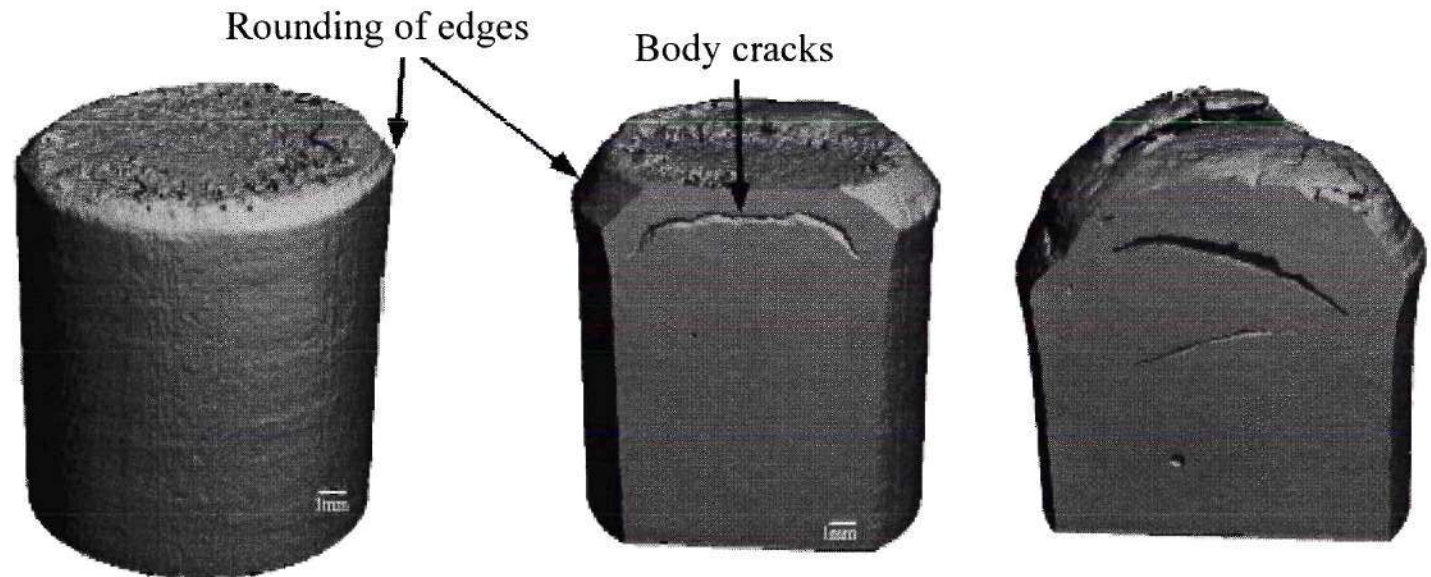
S.R. Stock, N.N. Naik, A.P. Wilkinson, and K.E. Kurtis, *Cement and Concrete Research*, October 2002, V.32:1673-5.

Microtomography

33,800 ppm sulfate
 Na_2SO_4 solution
Type I cement
 $w/c = 0.485$



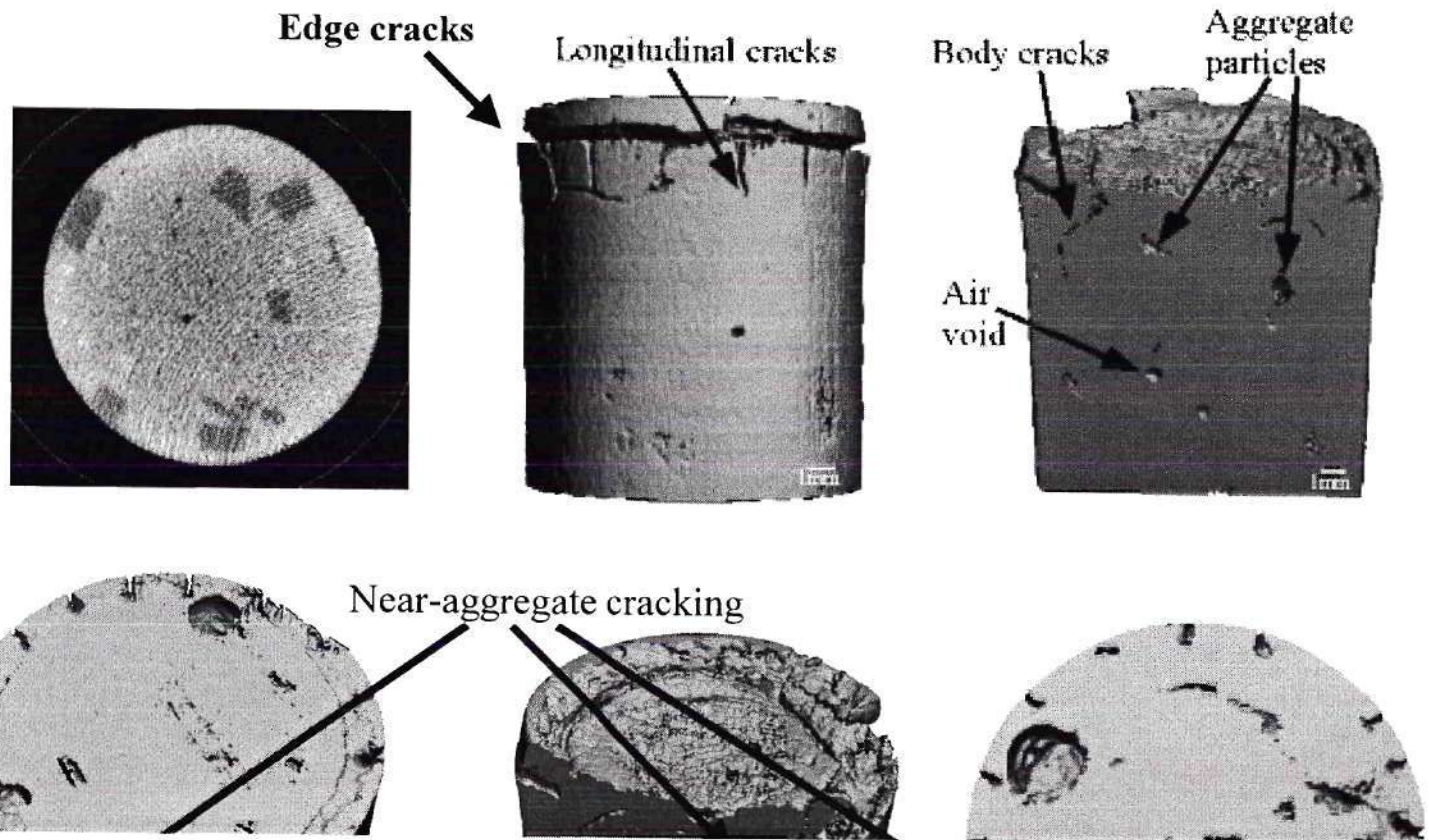
33,800 ppm sulfate
 MgSO_4 solution
Type I cement
 $w/c = 0.485$



Microtomography

10,000 ppm sulfate
 Na_2SO_4 solution
 Type I cement
 $w/c = 0.485$
 $\text{Agg}/c = 0.2$

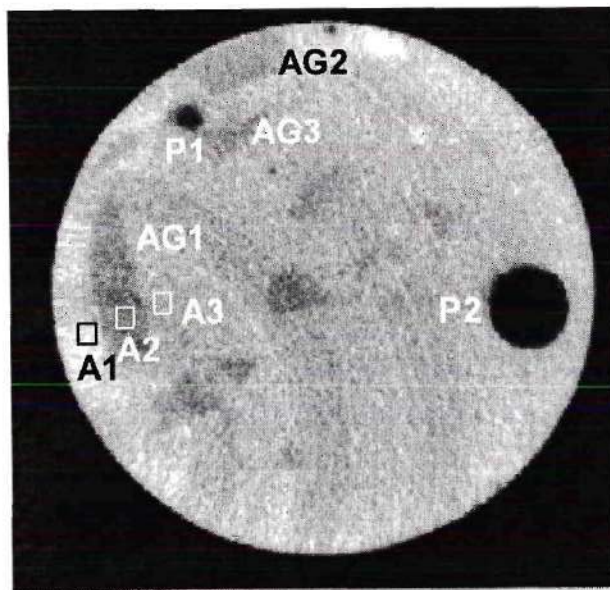
Aggregate is single
 quartz crystals from
 4.76mm (No. 4 sieve
 to 75 μm (No. 200
 sieve)



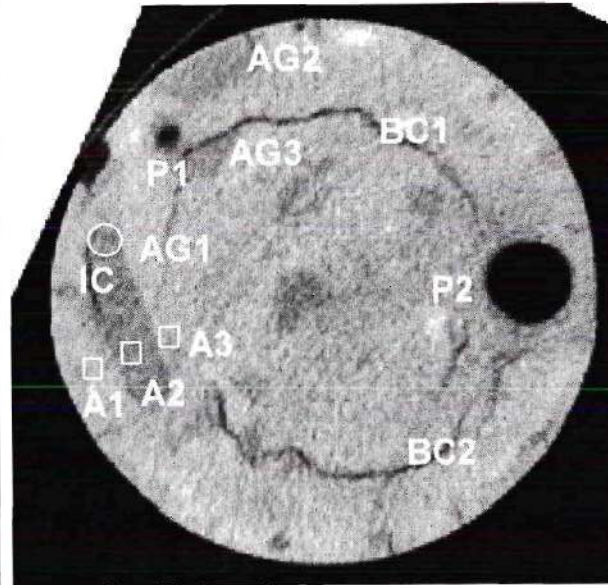
<u>Agg/c</u>	<u>edge cracks</u>	<u>body cracks</u>	<u>longitudinal surface cracks</u>
0	17 weeks	36 weeks	---
0.20	12 weeks	16 weeks	16 weeks

Microtomography

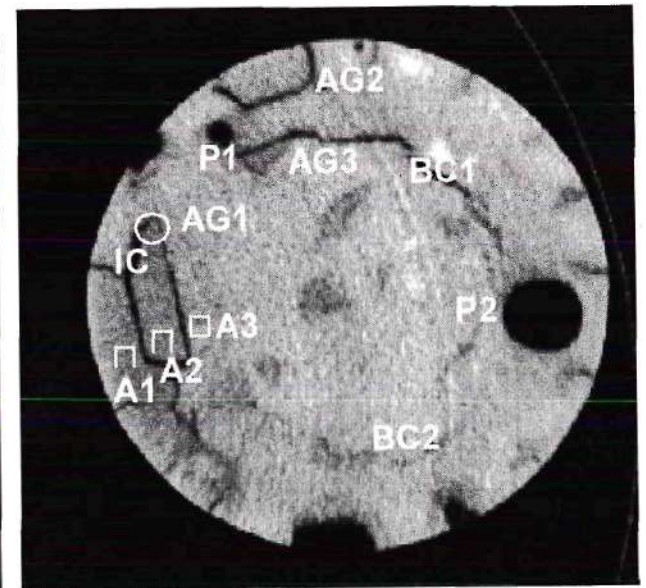
Type I cement paste-aggregate sample at $w/c = 0.485$ and exposed to 10,000 ppm of sulfate ions in sodium sulfate solution



7 weeks

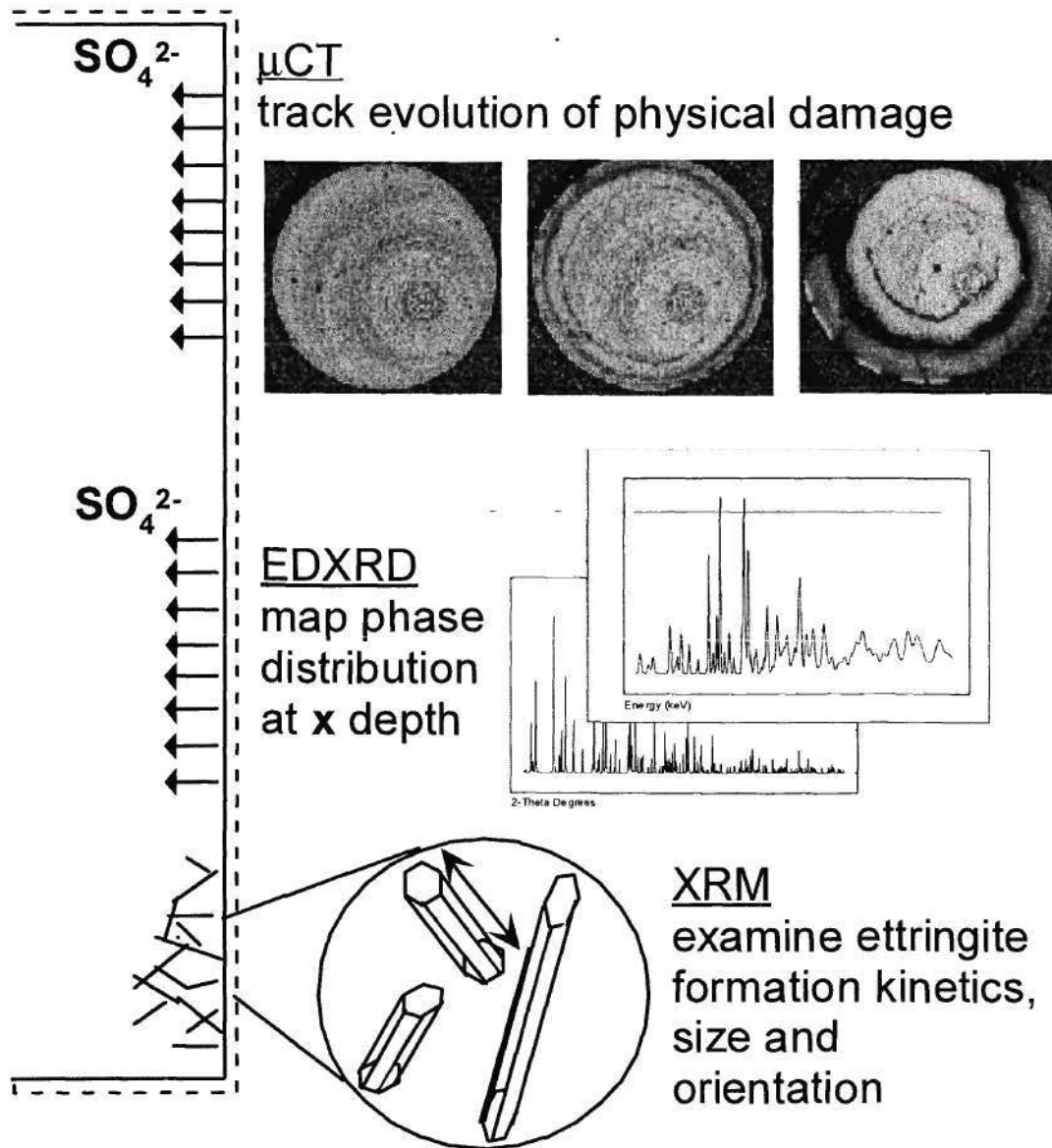


17 weeks

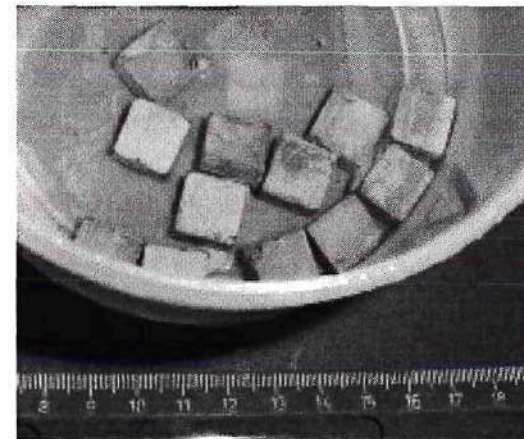


29 weeks

Integration of MicroCT Data



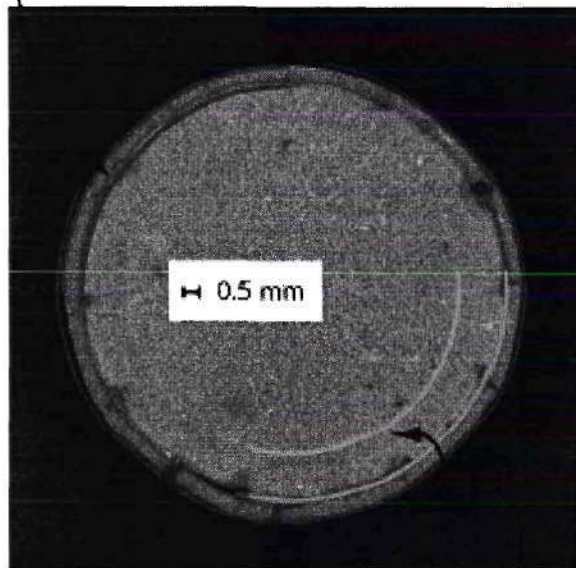
In addition to x-ray methods, project includes:
Mortar bar expansion measurements
Compressive strength measurements



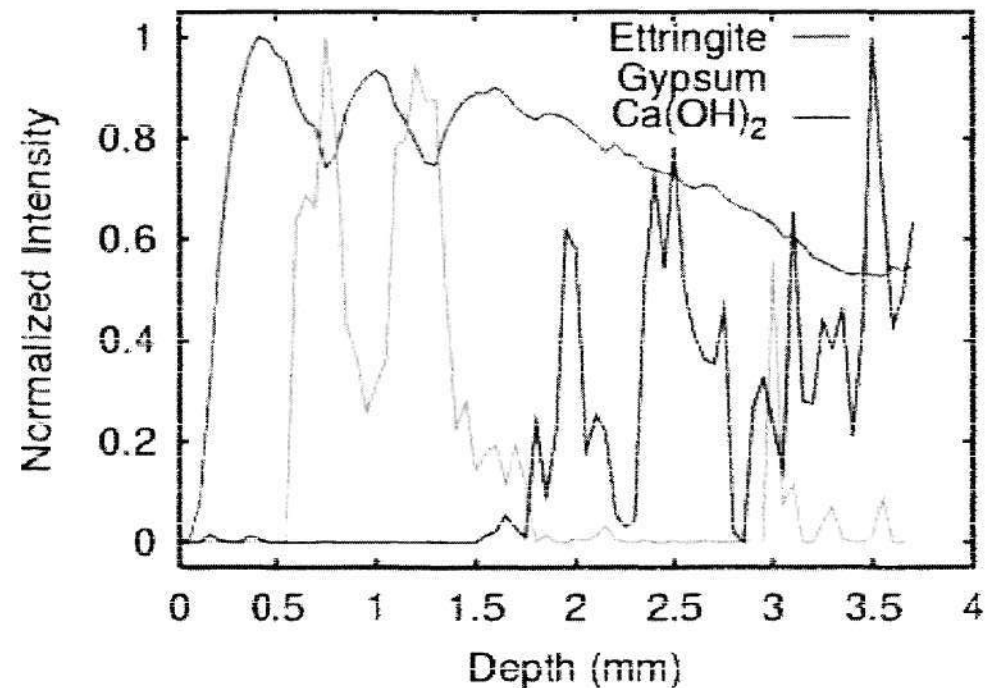
Microtomography and EDXRD

One focus of continuing work is improving spatial correlation between μ CT and EDXRD data

Ettringite-rich, gypsum-free layer
outside cylindrical crack



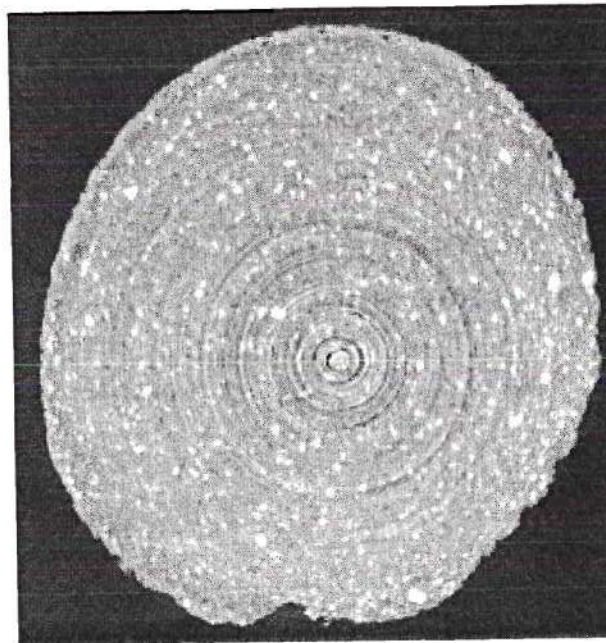
Gypsum-bearing region inside crack



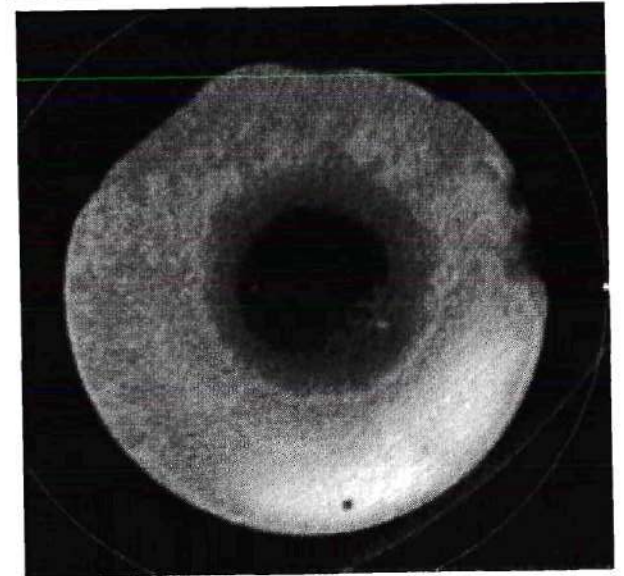
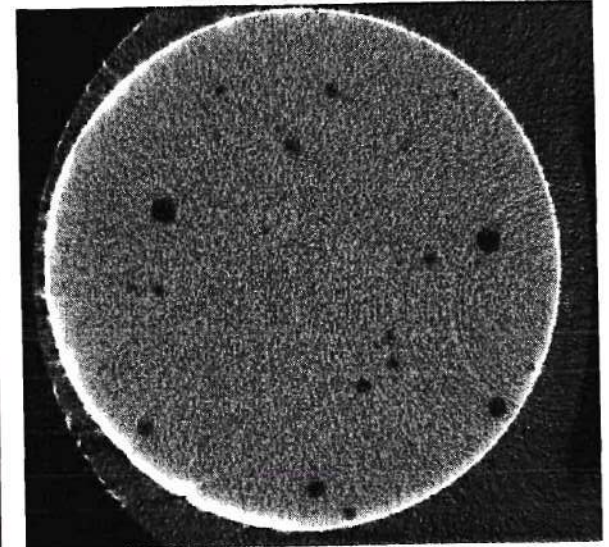
Microtomography

Another area of continuing work is the use of contrast agents to track ingress by:

- Synchrotron-based x-ray microtomography at APS
- Use of x-ray absorbing solutions to track ingress by lab-based microtomography



S.R. Stock, Northwestern University



Techniques

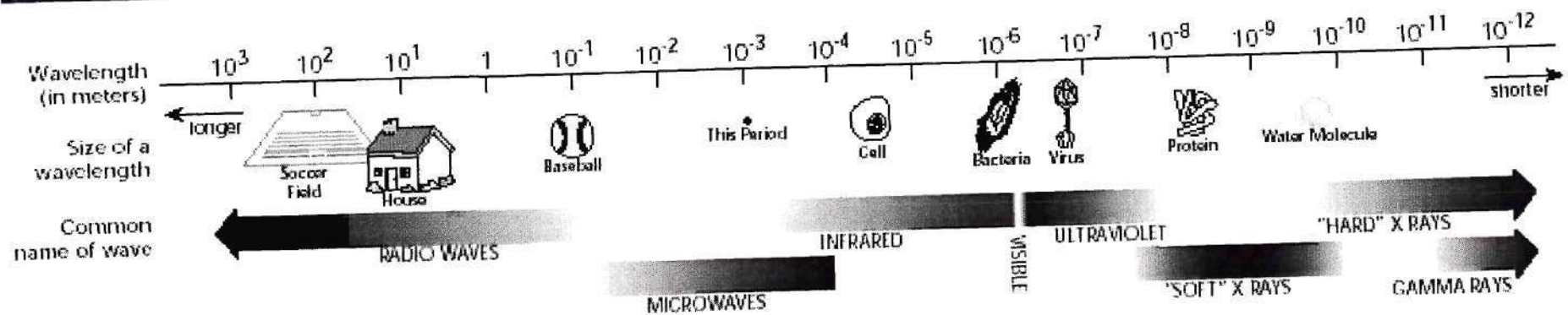
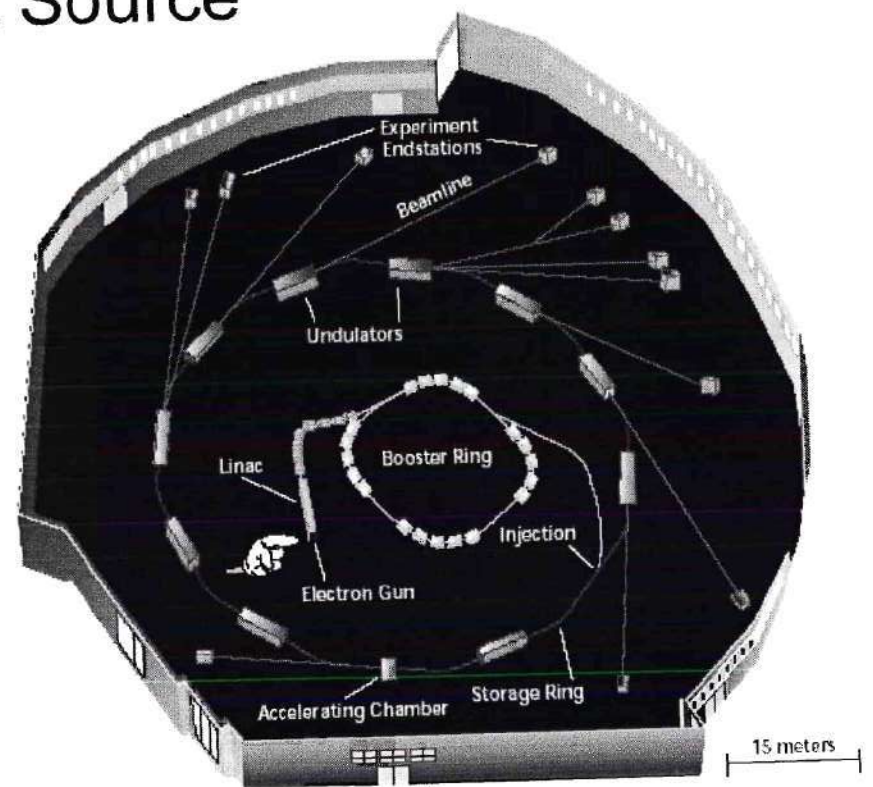
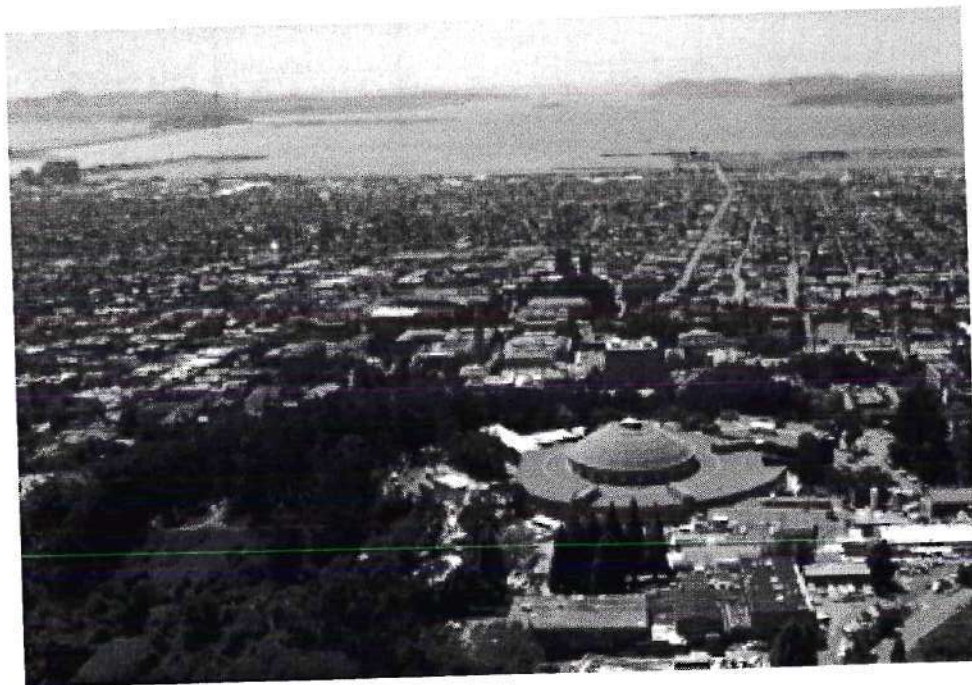
Transmission soft X-ray microscopy (TXRM)

Collaborators:

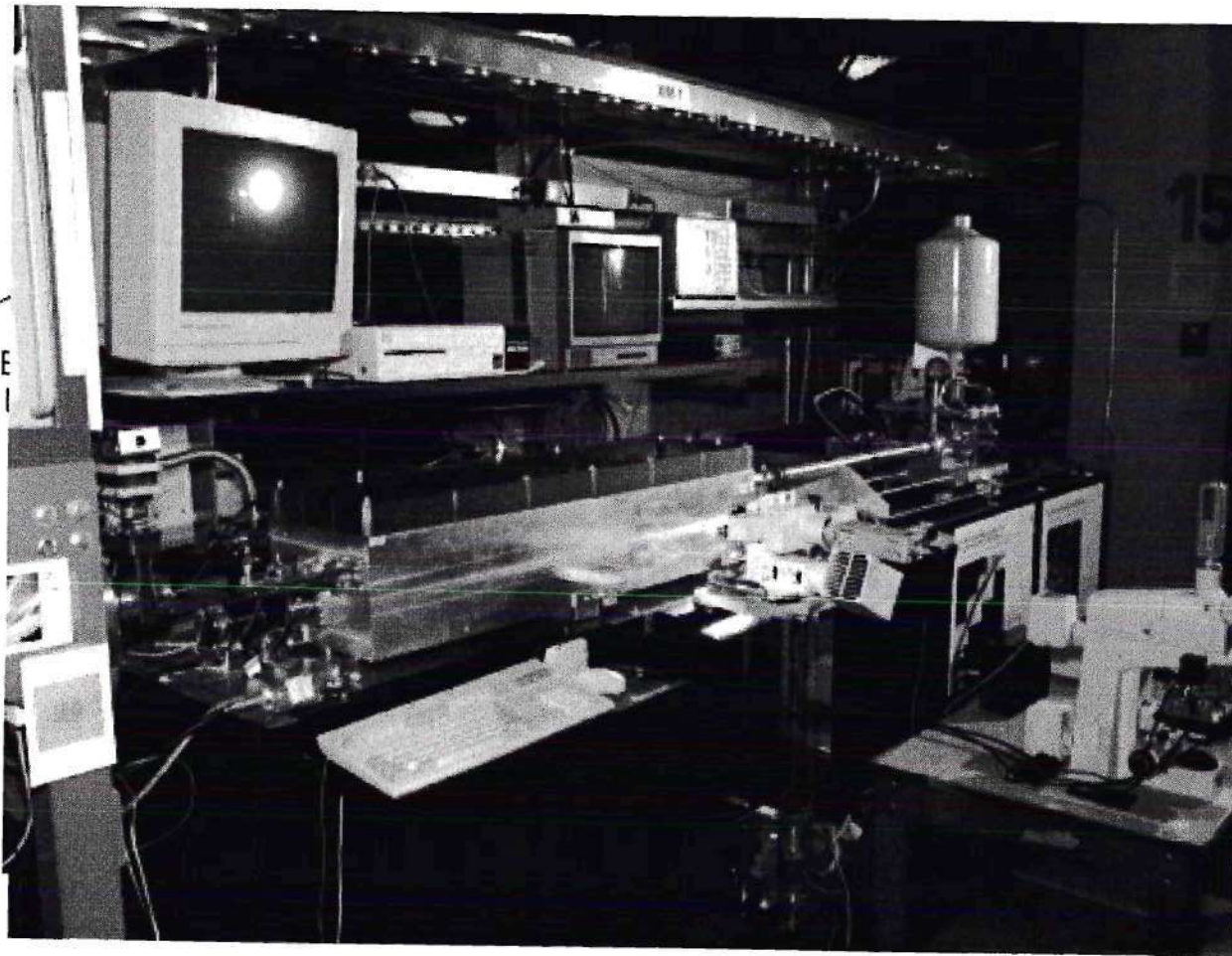
UC Berkeley: Paulo Monteiro

CXRO: Greg Denbeaux, Angelic Lucero

Advanced Light Source



XM-1: Beamline 6.1.2 at ALS



sensor zone plate
micro zone plate

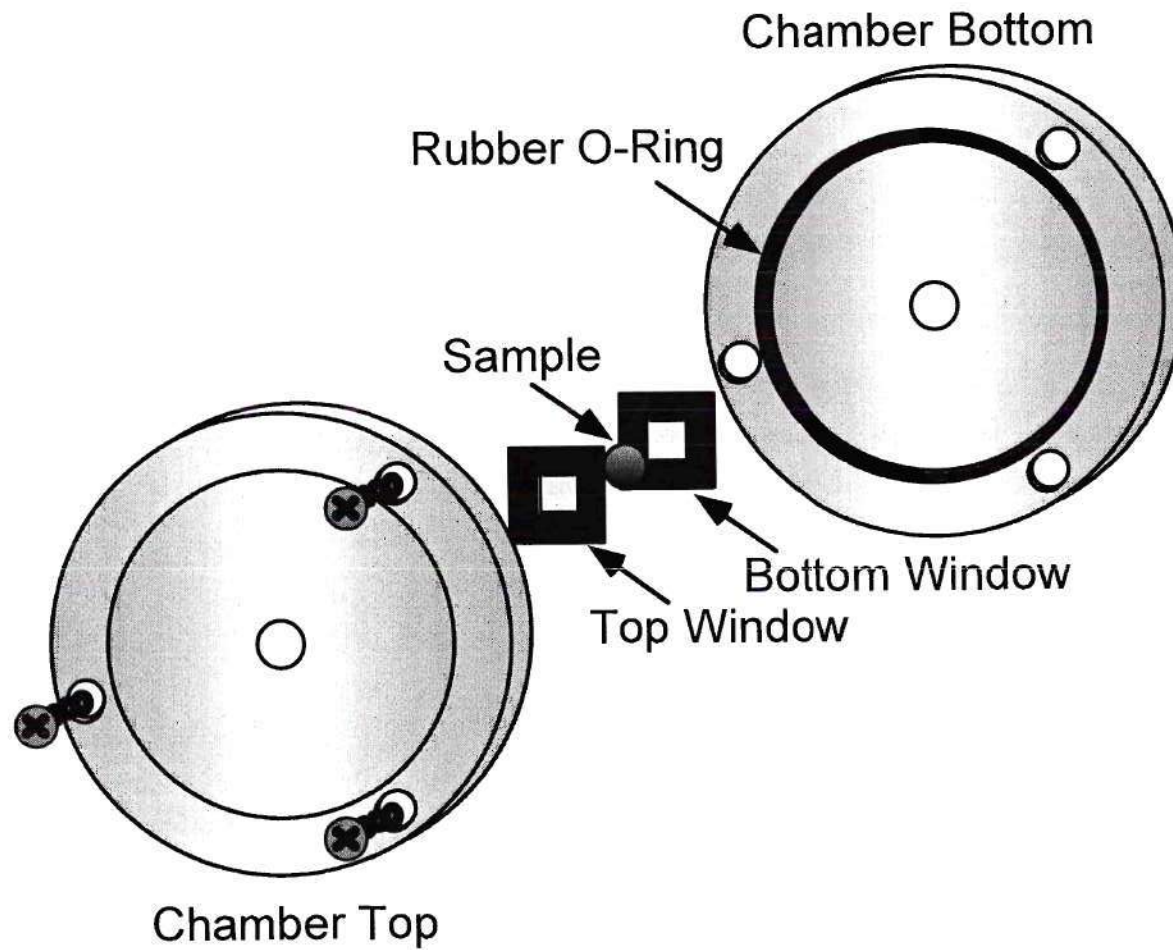
X-ray
CCD
camera

Light
scope



XM-1 is operated and maintained by the Center for X-ray Optics (CXRO)

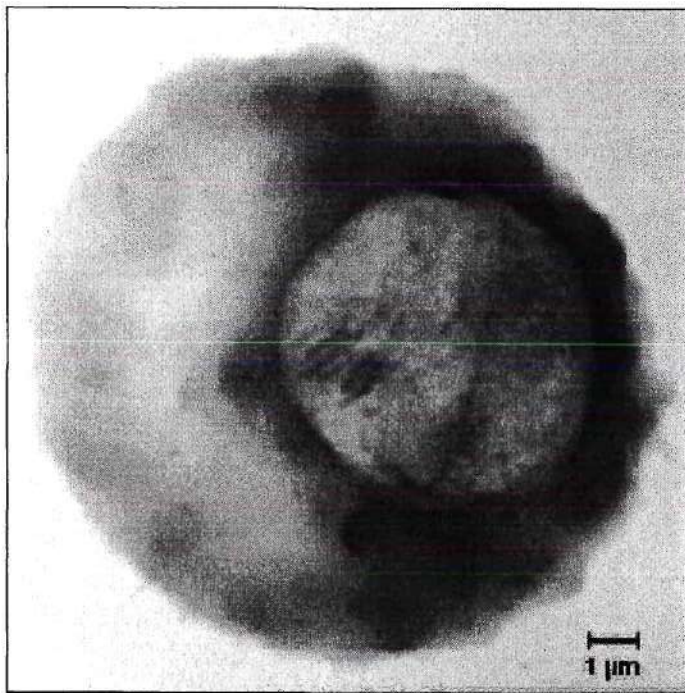
XM-1: Sample Preparation



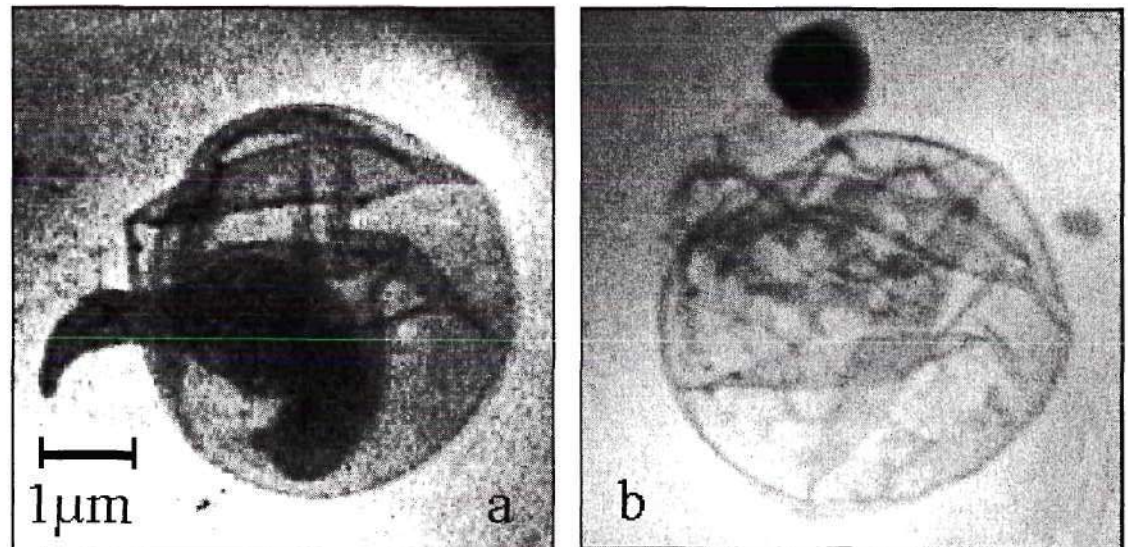
XM-1

Microscope originally developed to image biological samples.

- Imaging in wet environment
- Fast imaging times



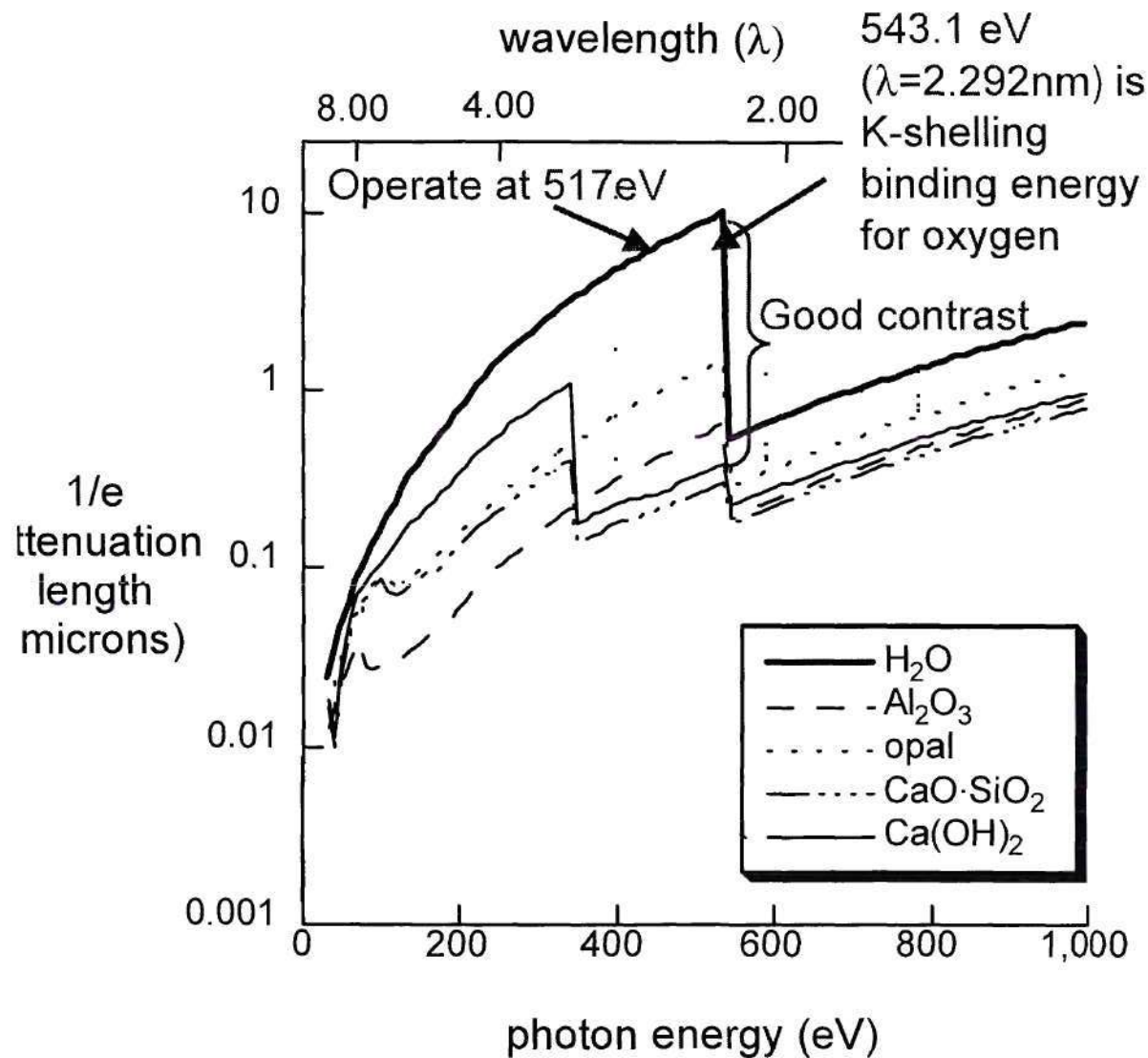
Malaria parasite in a human red blood cell
C. Magowan, LBNL



Cryptosporidium (a) sporozoite emerging from oocyst, (b) empty oocyst with residium

C. Petersen, UCSF, San Francisco General Hospital

XM-1



X-rays of wavelength 2.4 nm (517 eV), a wavelength just below the oxygen edge, are able to penetrate wet samples through several microns in depth.

At this wavelength, minerals such as alumina, opal, calcium silicates, and calcium hydroxide absorb nearly an order of magnitude more than water.

Thus, x-rays of 2.4nm wavelength are well-suited for studies of cement hydration and concrete deterioration products in wet environments.

$$E = \frac{hc}{\lambda}; E(\text{ev}) = \frac{1239.8}{\lambda(\text{nm})}$$

Transmission Soft X-ray Microscopy at XM-1

Advantages

- Designed for ease of user operation
- Samples can be studied wet
- No artifacts from drying or pressure change
- Able to observe and record ongoing reactions
- High resolution (43 nm)
- Characterization of internal structure
- Identify areas of elemental concentrations

Limitations

- Small sample size
- High solution-to-solid ratio needed for transmission
- Limited spectromicroscopy capabilities
- Limited availability

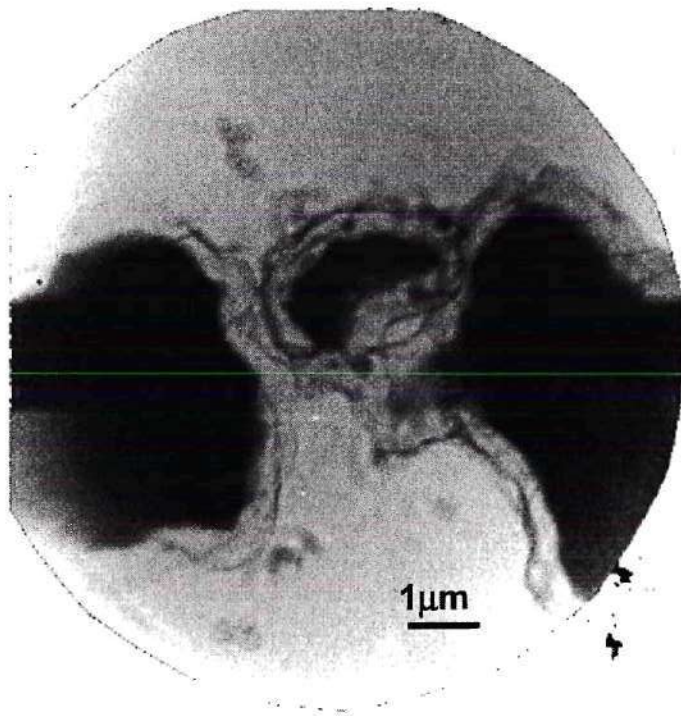
Transmission Soft X-ray Microscopy at XM-1

Some applications of TXRM relevant to microstructure and durability cement-based materials:

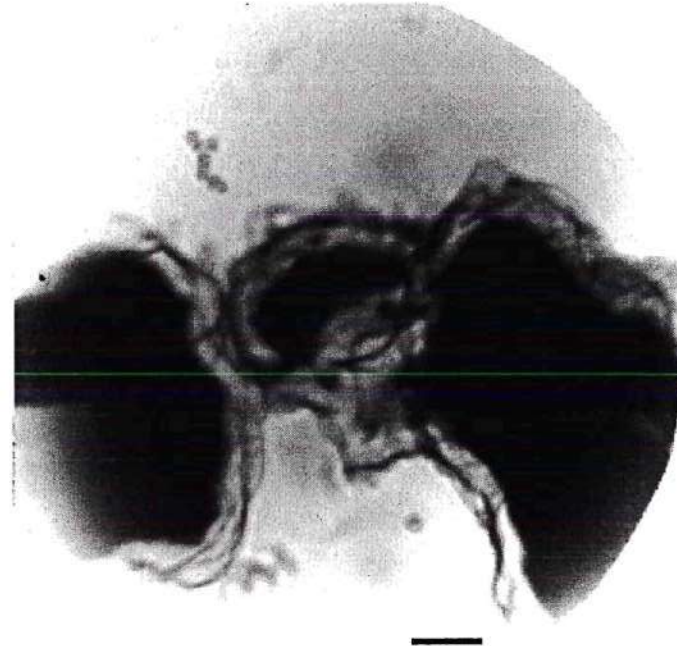
- Hydration of portland, rice hull ash, calcium sulfoaluminate, and calcium aluminate cements
- Pozzolanic reactions
- Alkali-silica reaction
- Sulfate attack
- Corrosion of steel

Cement Hydration

Examination of hydration of calcium sulfoaluminate cements
(Kleinite $C_4A_3\bar{S}$ or $4CaO \cdot 3Al_2O_3 \cdot \bar{SO}_4$)



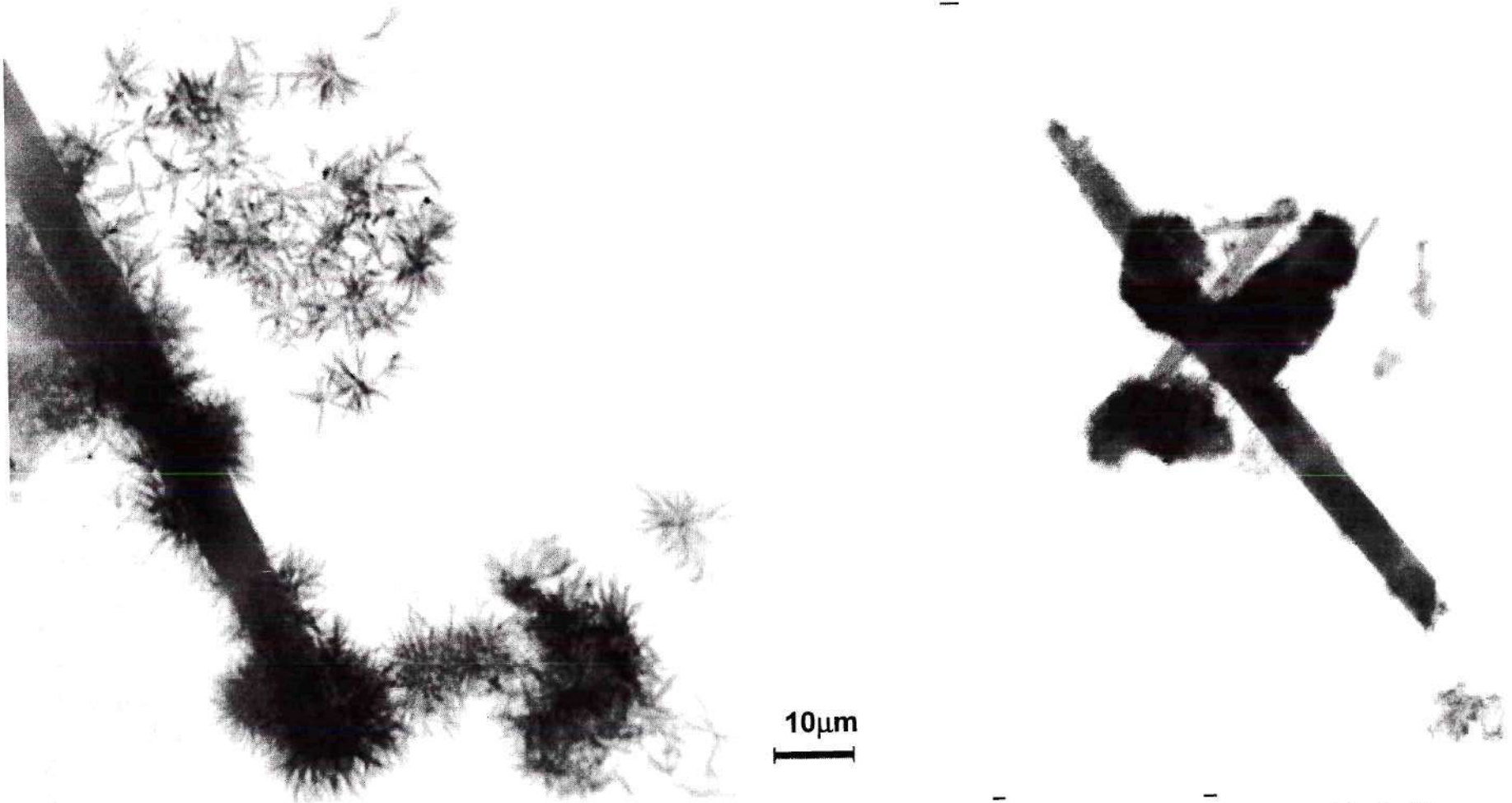
30 min.



85 min.

2400x; $\lambda=2.4nm$

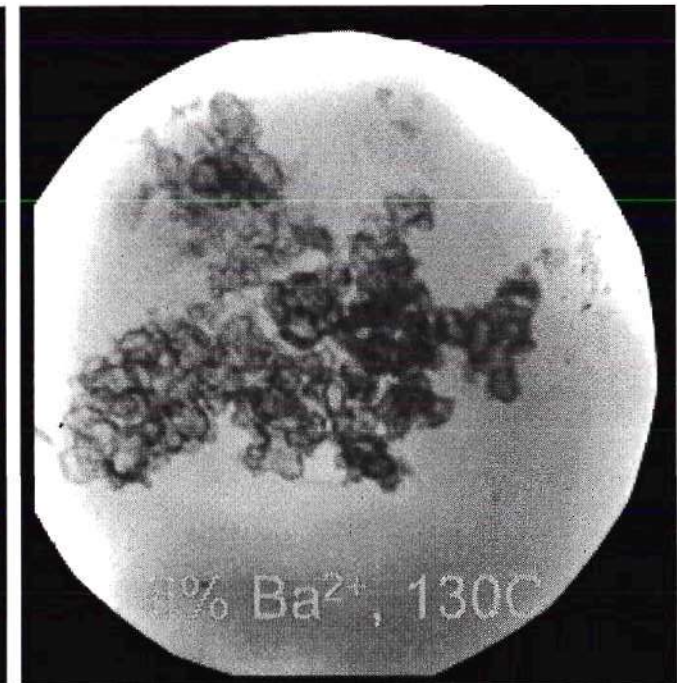
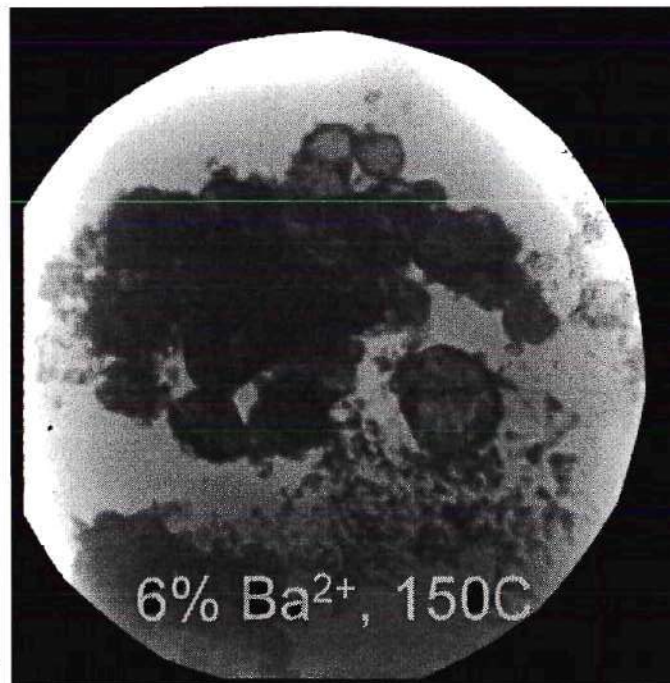
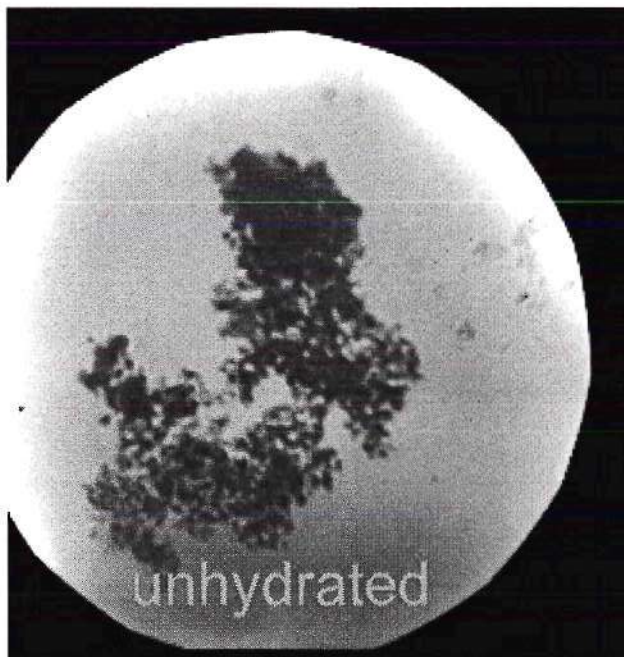
Cement Hydration



Tiles composed from X-ray images of mixture of 30.0% $C_4A_3\bar{S}$ + 53.4% $C\bar{S}$ + 16.5% C (by mass) after 4.5 hours in saturated calcium hydroxide + 0.1 M $CaCl_2$ solution, w/s=10

Cement Hydration

- Investigation of thermal treatment on hydration of low-energy, β - $2\text{CaO}\cdot\text{SiO}_2$ rice hull ash cements.
- RHA cements processed at higher temperatures (150°C vs. 130°C) reacted more rapidly, with products visible through XRM at earlier ages and with greater abundance.



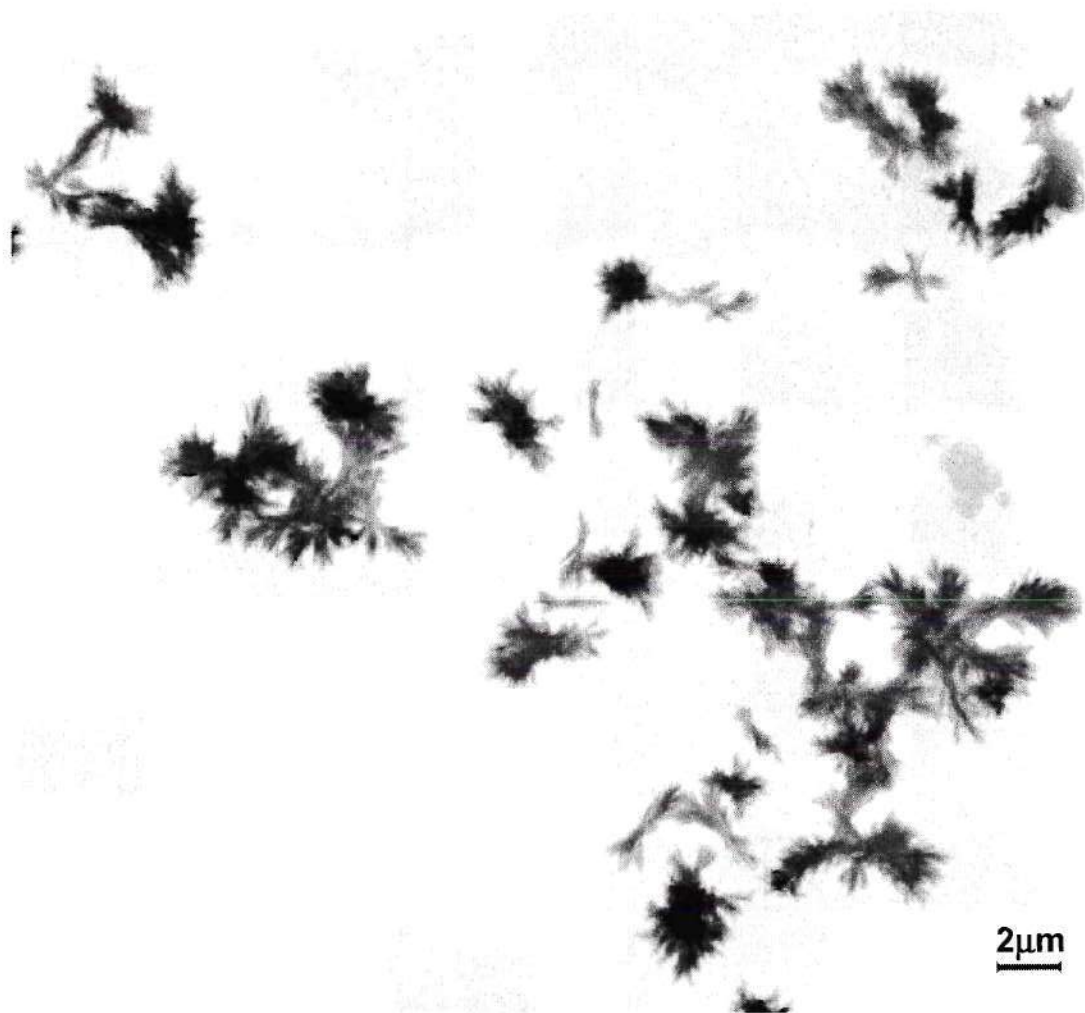
2400x; $\lambda=2.4\text{nm}$

K.E. Kurtis and F.A. Rodrigues, *Cement and Concrete Research*, April 2003, V.33(4):509-515

Pozzolanic Reaction

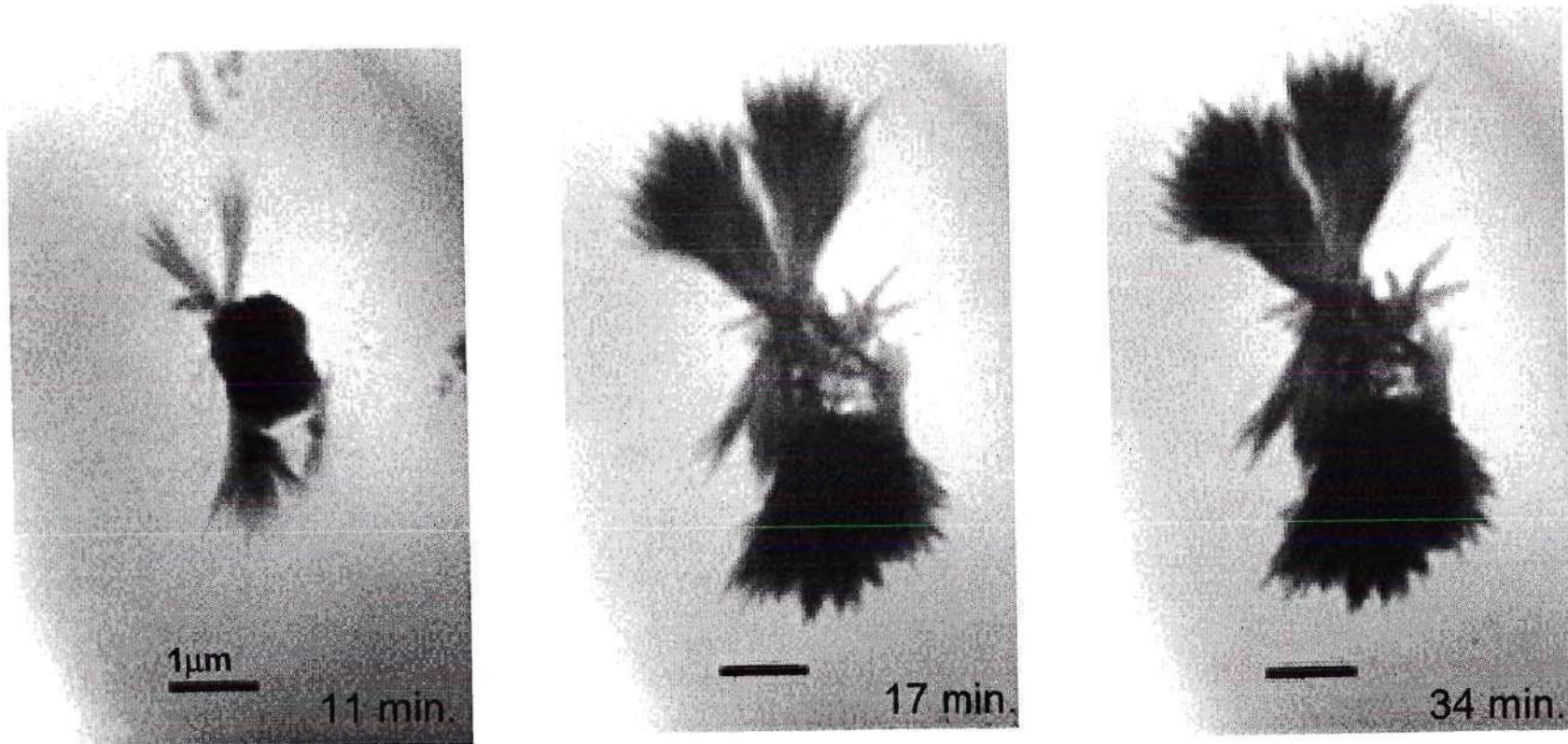
Reaction of finely divided silicates in alkaline calcium-rich solutions generally resulted in products with dendritic microstructure

The 'sheaf of wheat' morphology was particularly prevalent



Tiled x-ray images of chemical grade silica gel after 2 hours in saturated $\text{Ca}(\text{OH})_2$ solution.

Pozzolanic Reaction

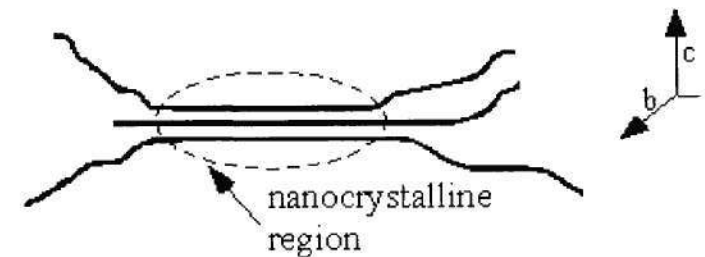
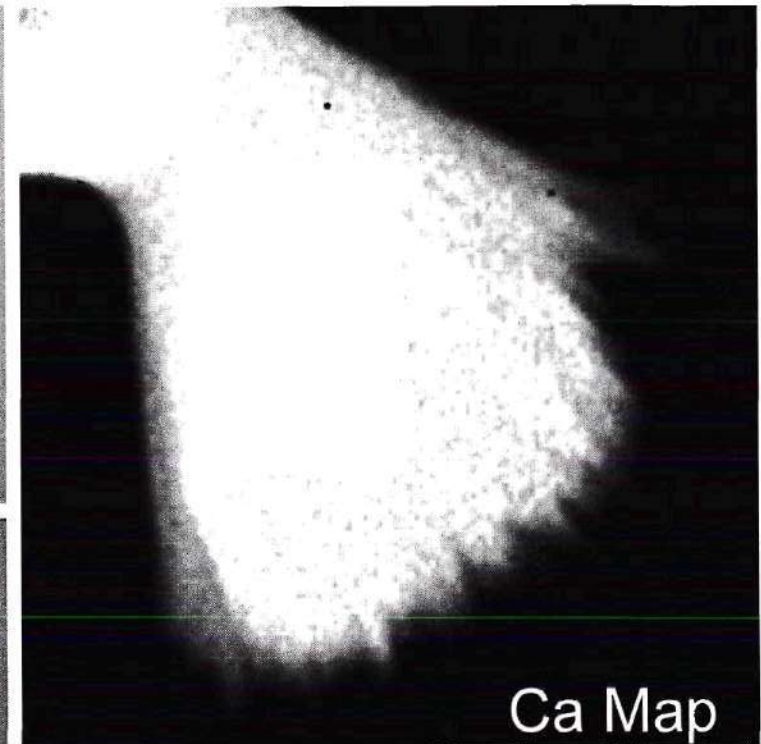
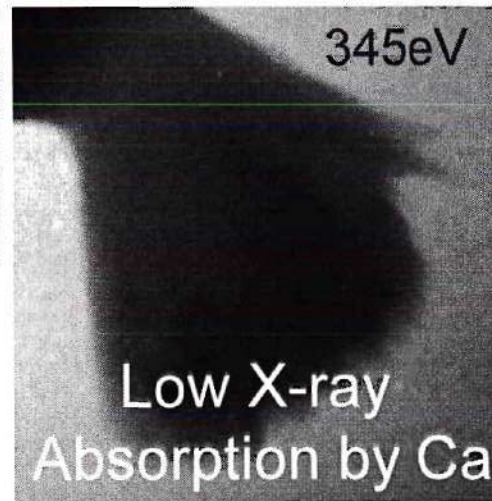
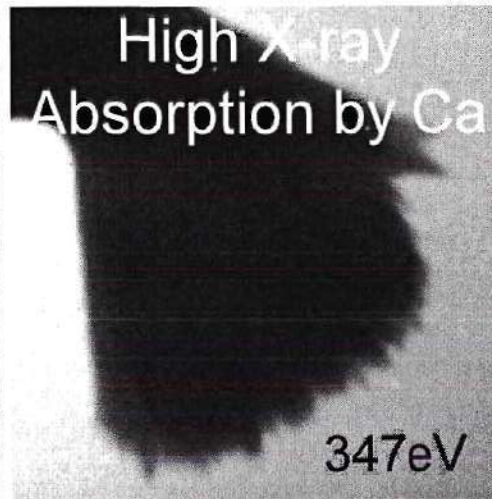


Reaction of silica gel in 0.7M NaOH + 0.1M CaCl₂ solution over time results in development of “sheaf of wheat” microstructure.

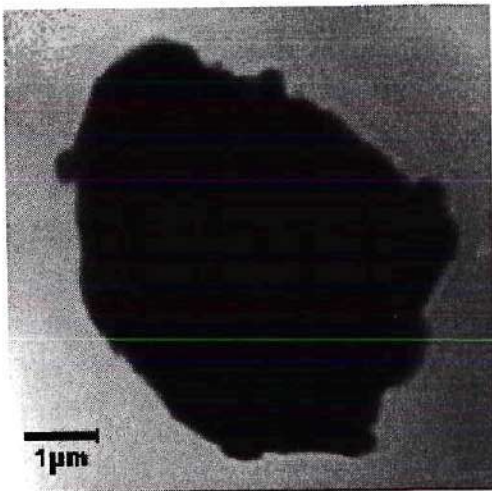
K.E. Kurtis, P.J.M. Monteiro, J.T. Brown, and W. Meyer-Illse, *Journal of Microscopy*, December 1999, V196: 288-298.

E. Gartner, K.E. Kurtis, and P.J.M. Monteiro, *Cement and Concrete Research*, May 2000, V30 (5):817-822. 2400x; $\lambda=2.4\text{nm}$

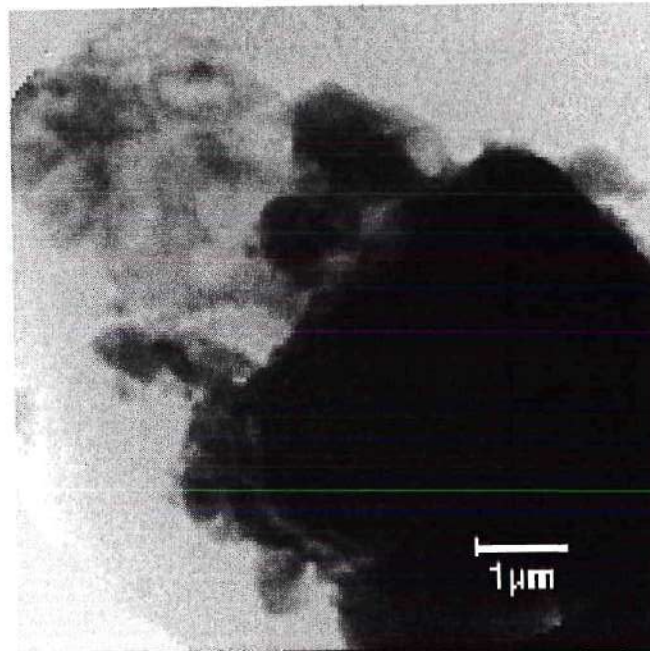
Pozzolanic Reaction: Ca-edge Spectromicroscopy



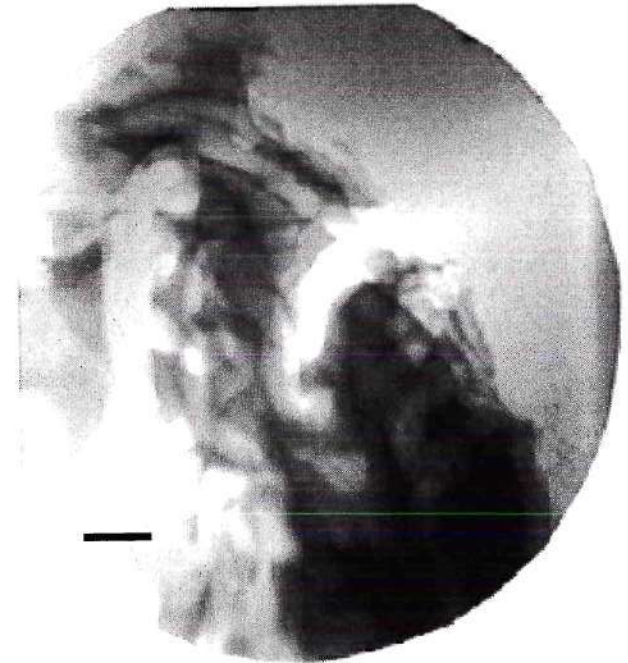
Alkali-Silica Reaction



“Dry” ASR gel;
Not in solution



ASR gel in 0.05M NaOH
at pessimum proportion



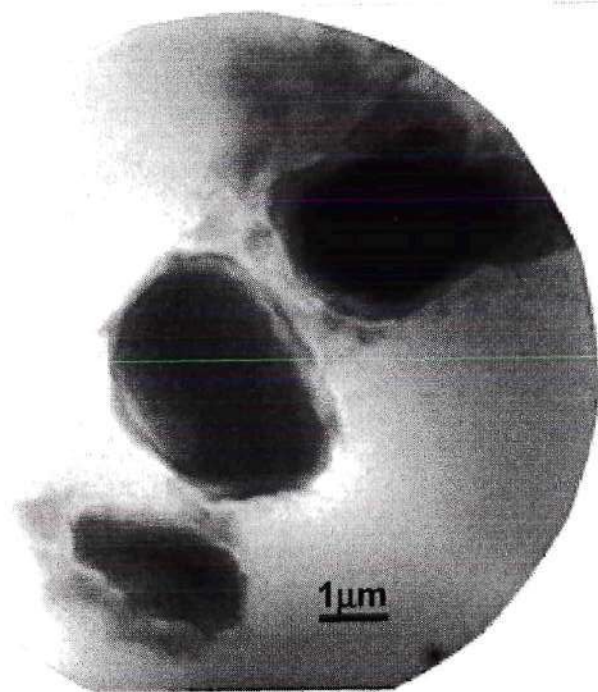
ASR gel in 0.7M NaOH at
pessimum proportion

K.E. Kurtis, P.J.M. Monteiro, J.T. Brown, and W. Meyer-Ilse, *Journal of Microscopy*, December 1999, V196: 288-298.

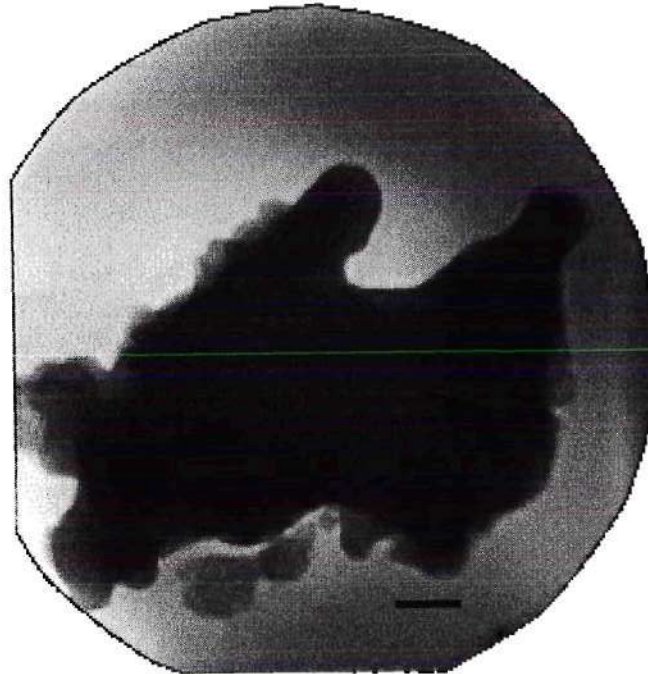
K.E. Kurtis, P.J.M. Monteiro, J.T. Brown, and W. Meyer-Ilse, *Cement and Concrete Research*, March 1998, V28:411-421.

Alkali-Silica Reaction

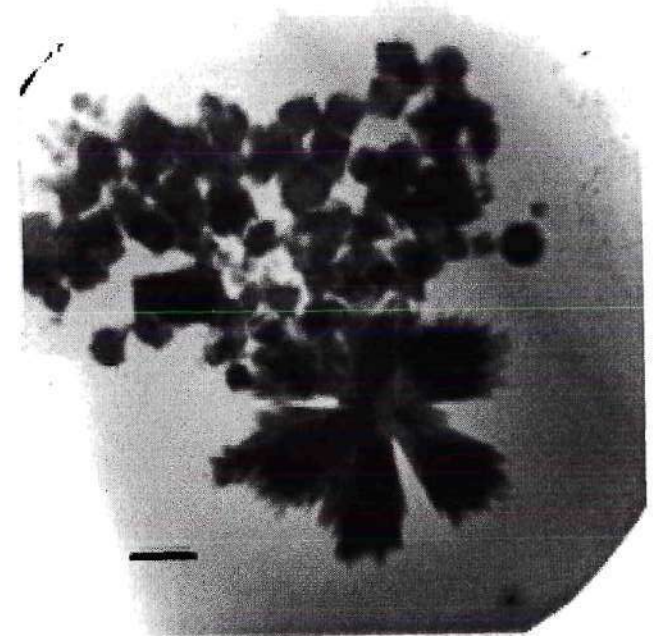
Examination of the effect of “expansion-controlling” chemical additives, including acetone and lithium chloride



After 1 week in 0.7M NaOH
+ 10% acetone



After 1 week in 0.7M NaOH + 0.1M LiCl



Techniques

Laser scanning confocal microscopy (LSCM)

Collaborators:

Ph.D. Students: Nabil El-Ashkar, Nikhila Naik, Ben Mohr

M.S. Thesis Student: Courtney Collins

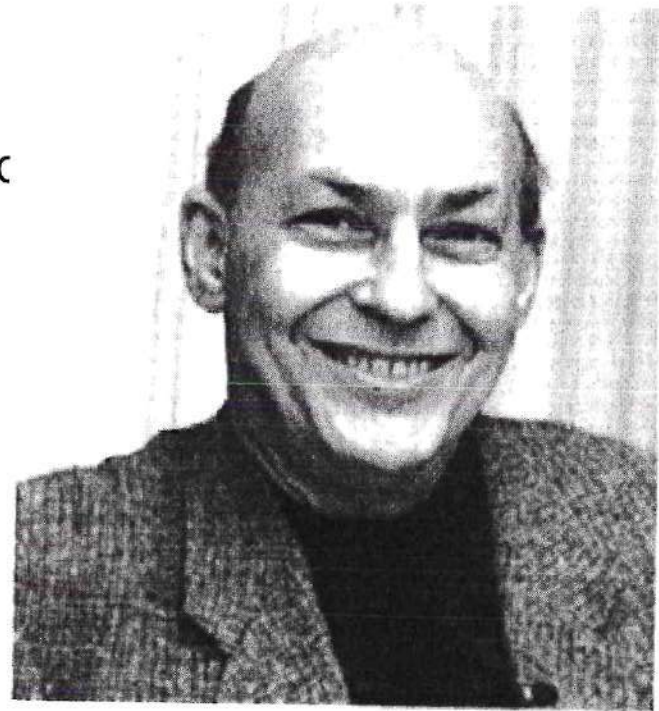
REU Students: Jason Ideker, Gayle Willis

Confocal Microscopy

The working principle of the confocal microscope is based upon a patent for “a double-focusing stage scanning microscope”, filed in 1957 by Marvin Minsky (now at MIT), while at Harvard.

Other Minsky inventions:

- The SNARC, the first neural network simulator
- "Muse" synthesizer
- LOGO "turtle"
- Mechanical hands



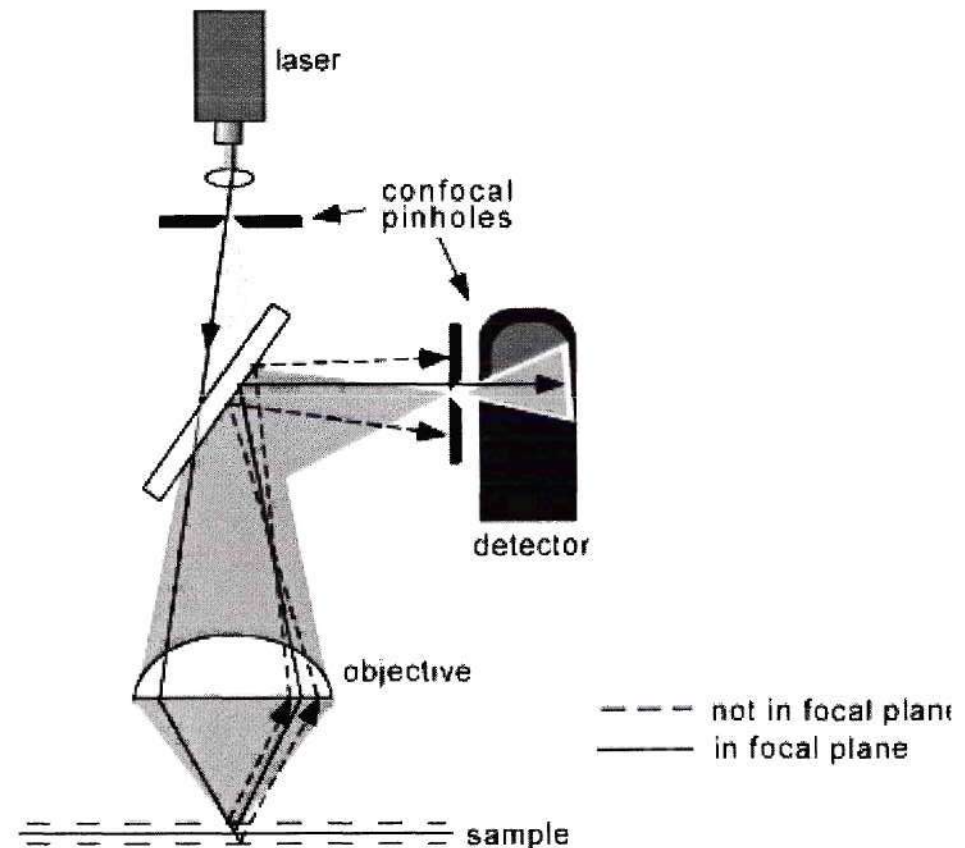
Marvin Minsky

Toshiba Professor of Media Arts & Sciences
Professor of E.E. and C.S., M.I.T

Laser Scanning Confocal Microscopy

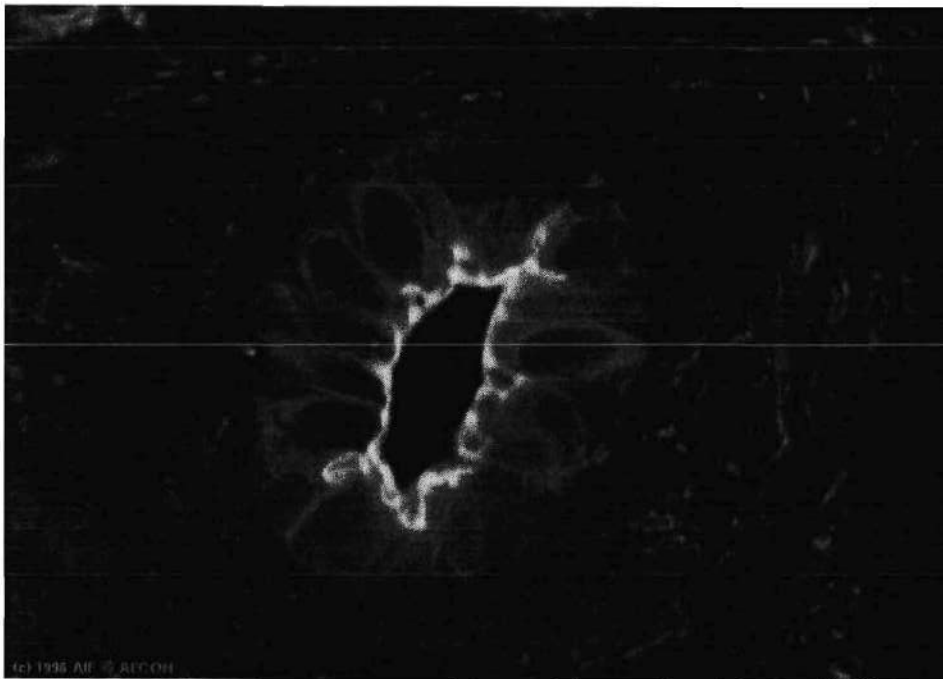
Three-dimensional representations are formed by acquiring a series of images of the same object at consecutive focal planes.

- A series of diaphragms focus light supplied by a laser onto the sample.
- Only one location on the sample is illuminated at a time.
- The reflected and fluorescent light passes through a point detector which discards rays that are reflected or fluoresced by planes which are not in focus, yielding enhanced axial resolution and the ability to perform non-invasive optical sectioning
- The resulting images are essentially slices of the sample taken sequentially deeper (or shallower) through the sample.



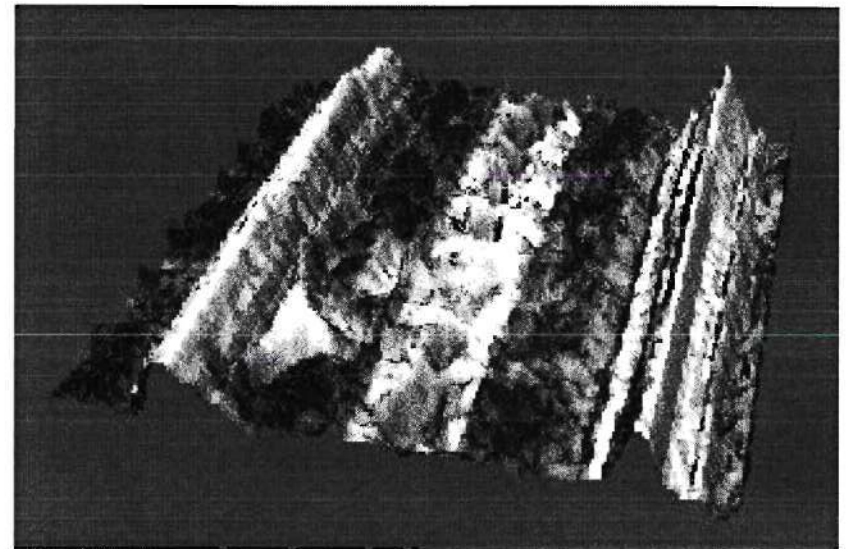
Laser Scanning Confocal Microscopy

LSCM is widely used in many fields including materials science, textile engineering, and biology, but its use to characterize cement-based materials remains relatively unexplored.



Confocal image of a slice of rat liver, treated with 3 fluorescent dyes

P. Novikoff Department of Pathology, AECOM



3D rendering of height profile of light emitting porous silicon (LEPSi) wafer in reflected-light mode

S. Damaskinos and A.E. Dixon, Department of Physics, Univ. Waterloo

Laser Scanning Confocal Microscopy

Advantages

- Samples can be studied wet
- No artifacts from drying or pressure change
- Able to observe and record ongoing reactions
- Good resolution
- Wide range of magnification
- Volumetric characterization of structure
- Able to image rough surfaces
- Can perform fluorescence microscopy to isolate microstructural features

Limitations

- Optical sectioning limited to transparent materials
- Contrast can be poor
- Fluorescence microscopy of cement-based materials is not well-developed

LSCM for Characterization of Cement-Based Materials

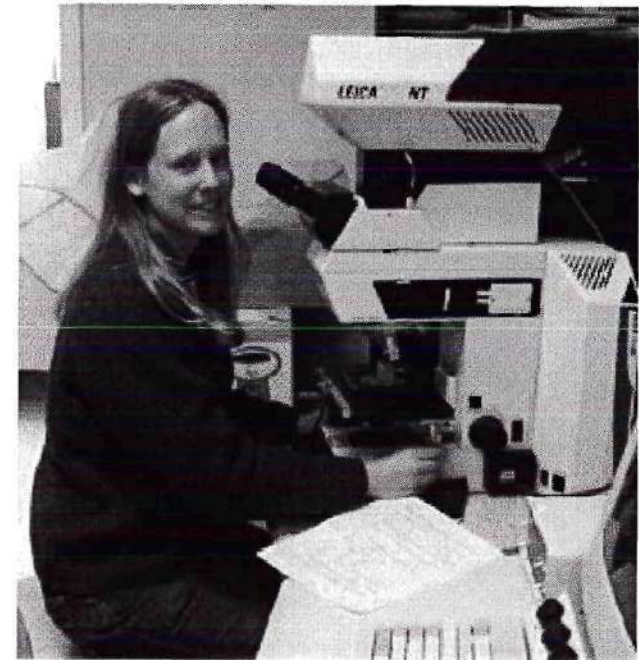
A Leica TCS NT Ar-laser confocal has been configured specifically for examination of cement-based materials. Some key features include:

- Multiple objectives allow for broad 2.5-2000x magnification range (micro → mesostructure)
- Prefer water immersion objectives for examining wet mounts
- Some objectives are cover-glass corrected (to ~2mm)
- Use of several sample stages allow for flexibility in sample size and type
- Indexable stage
- Two detectors allow for operation in reflected light mode, transmitted light mode, or both modes simultaneously
- Ar laser permits fluorescence microscopy between 488 and 514 nm

Laser Scanning Confocal Microscopy

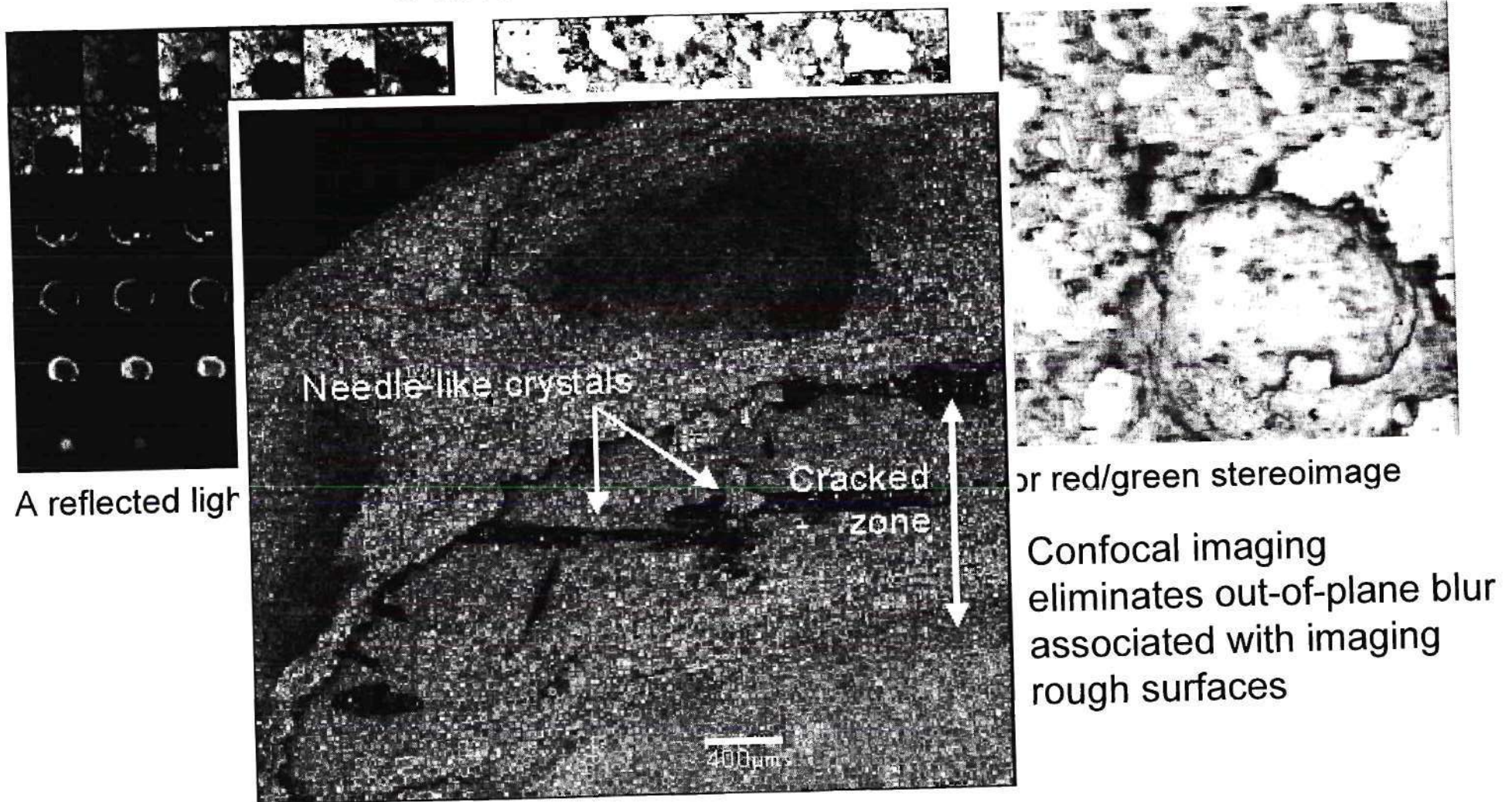
The application of four LSCM imaging methodologies to cement-based materials will be described. These are:

- (1) surface characterization
- (2) wet-chemistry studies, and
- (3) “through-aggregate” examination
- (4) fluorescence microscopy



This material is based upon work supported by the National Science Foundation under POWRE Award CMS-0074874. Any opinions, findings, and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the National Science Foundation.

Surface Characterization



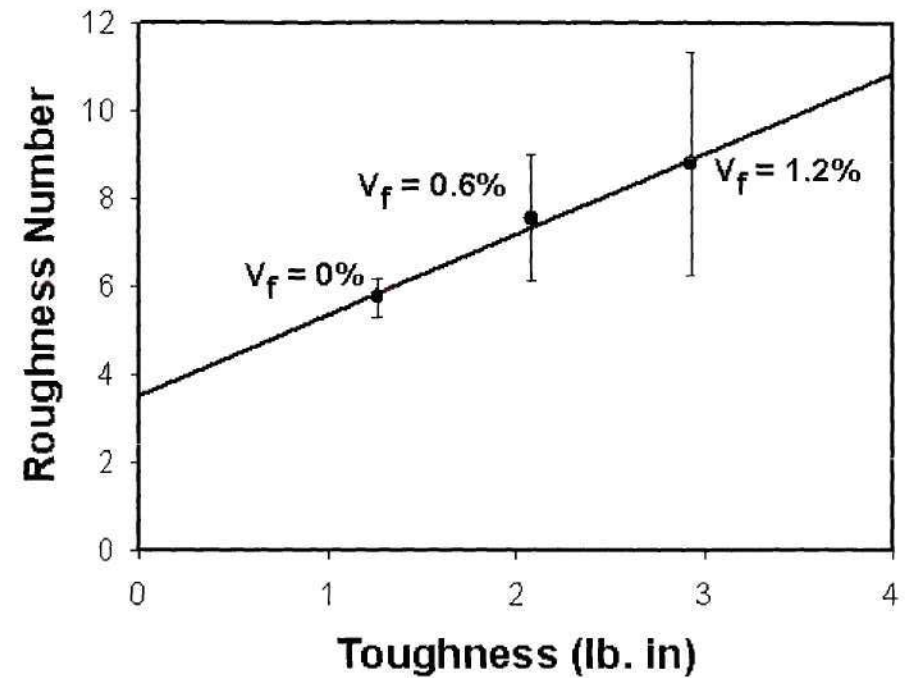
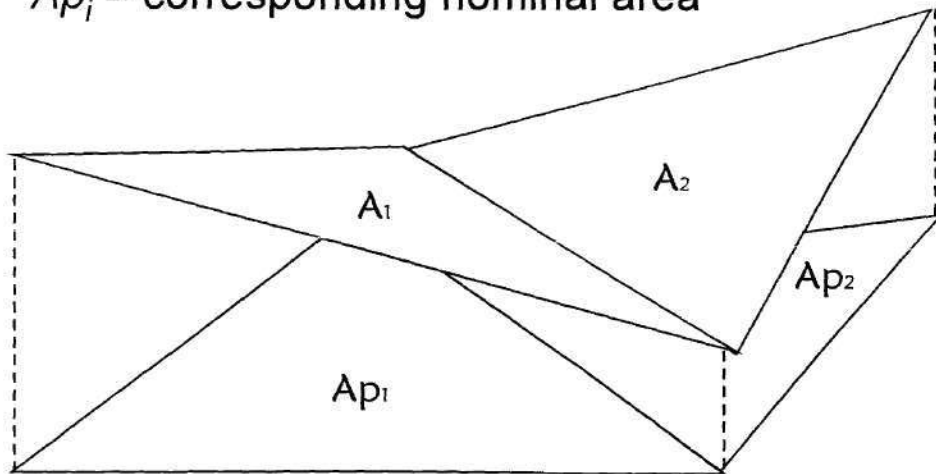
K.E. Kurtis, N.H. El-Ashkar, C.L. Collins, and N.N. Naik, "Examining Cement-Based Materials by Laser Scanning Confocal Microscopy", *Cement & Concrete Composites*, in press.

Surface Characterization

$$RN = \frac{\sum A_i}{\sum Ap_i}$$

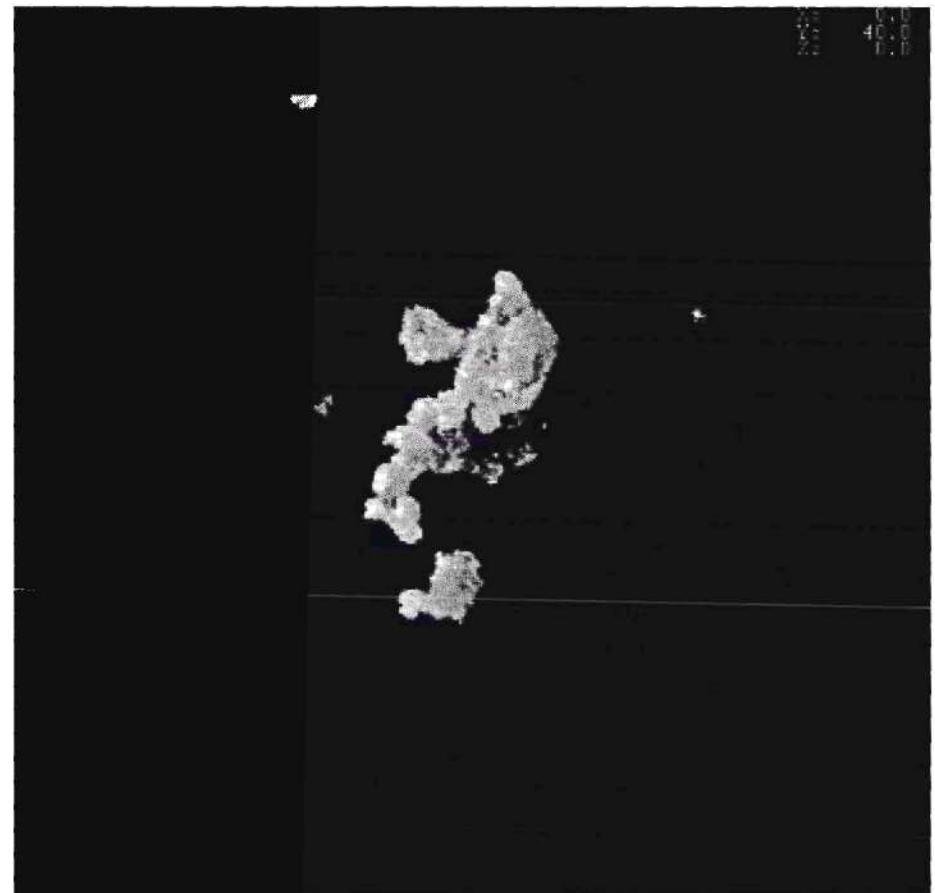
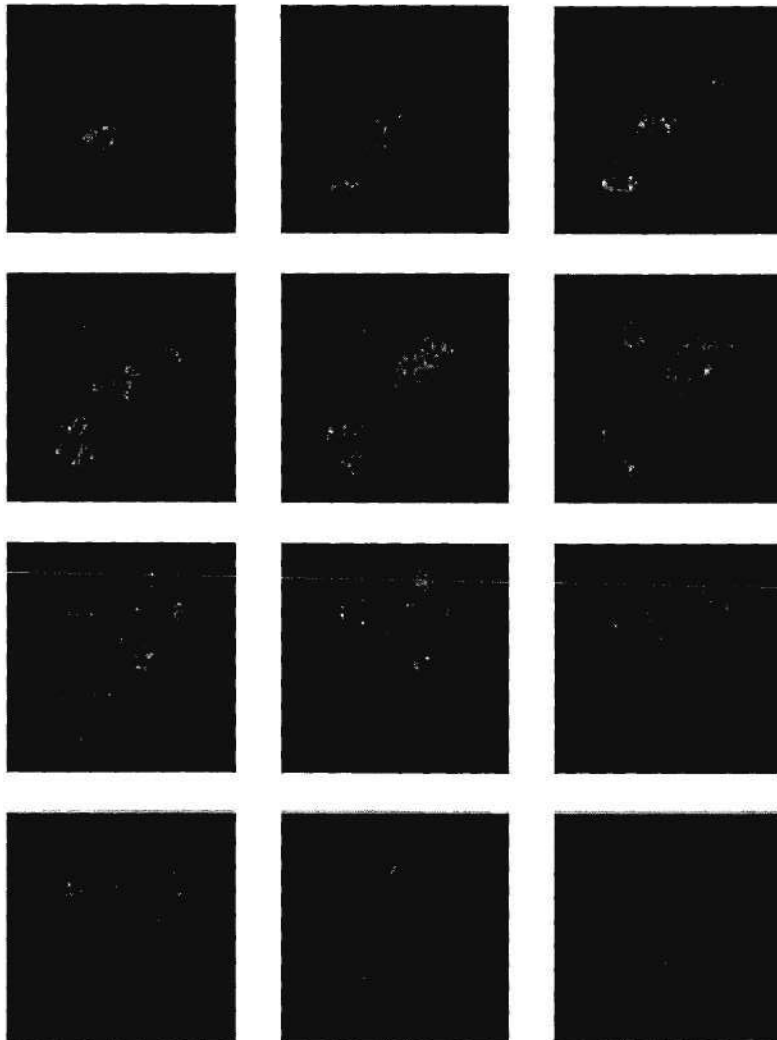
A_i = triangulated surface

Ap_i = corresponding nominal area



K.E. Kurtis, N.H. El-Ashkar, C.L. Collins, and N.N. Naik, "Examining Cement-Based Materials by Laser Scanning Confocal Microscopy", *Cement & Concrete Composites*, in press.

Wet Chemistry



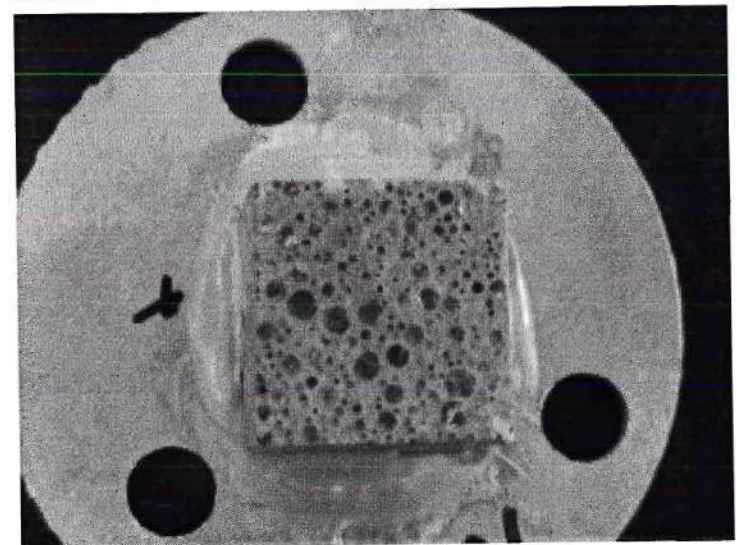
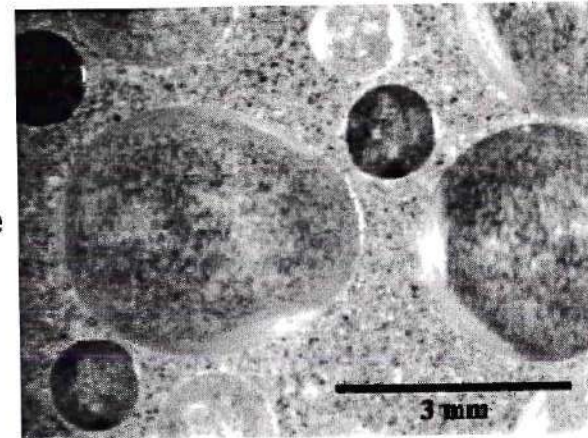
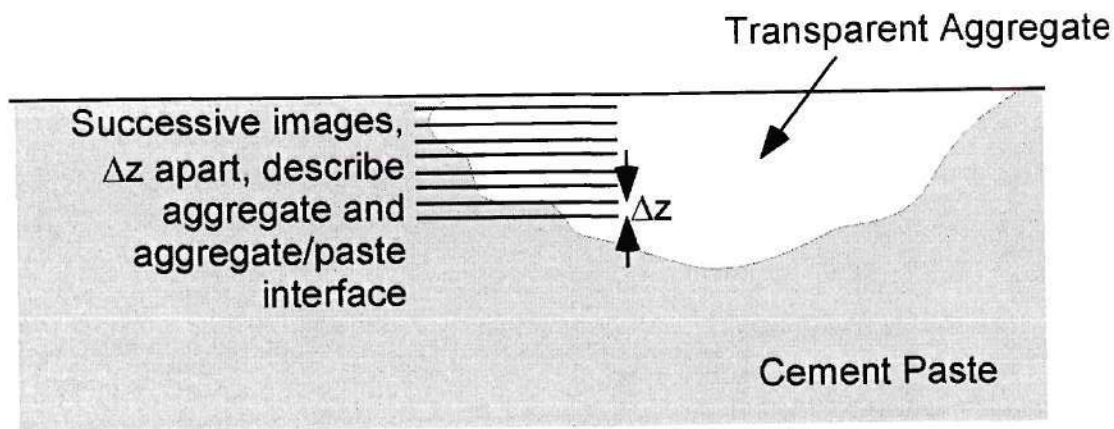
Sheaf of wheat microstructure resulting from pozzolanic reaction

K.E. Kurtis, N.H. El-Ashkar, C.L. Collins, and N.N. Naik, "Examining Cement-Based Materials by Laser Scanning Confocal Microscopy", *Cement & Concrete Composites*, in press.

Through-Aggregate LSCM

“Optical sectioning limited to transparent materials”

- Can be used advantageously



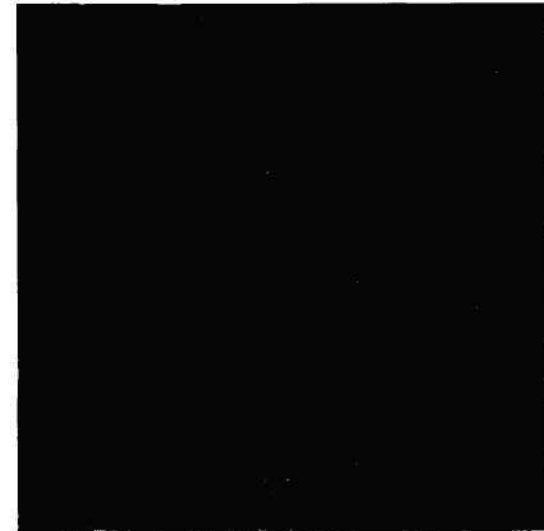
K.E. Kurtis, N.H. El-Ashkar, C.L. Collins, and N.N. Naik, "Examining Cement-Based Materials by Laser Scanning Confocal Microscopy", *Cement & Concrete Composites*, in press.

C.L. Collins, J.H. Ideker, and K.E. Kurtis, "Laser Scanning Confocal Microscopy for In Situ Monitoring of Alkali-Silica Reaction", *Journal of Microscopy*, accepted for publication.

C.L. Collins, J.H. Ideker, G.S. Willis, and K.E. Kurtis, "Examination of the Effects of LiOH, LiCl, and LiNO₃ on Alkali-Silica Reaction", submitted to *Cement and Concrete Research*.

Through-Aggregate LSCM

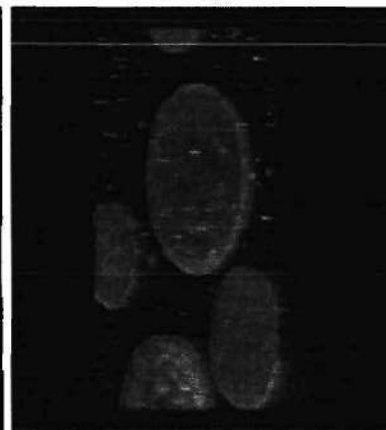
3-D reconstructions from confocal images obtained spherical through polished glass aggregate embedded in cement paste



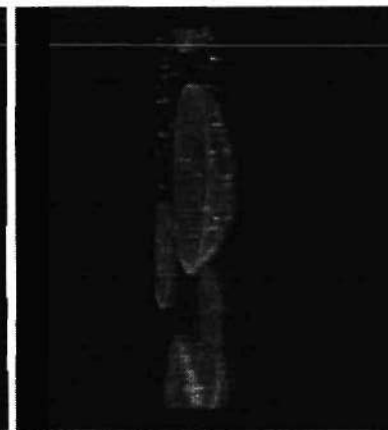
(1) x-y plane



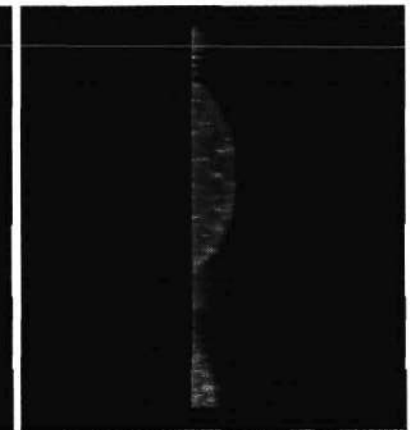
(2) rotation



(3) rotation

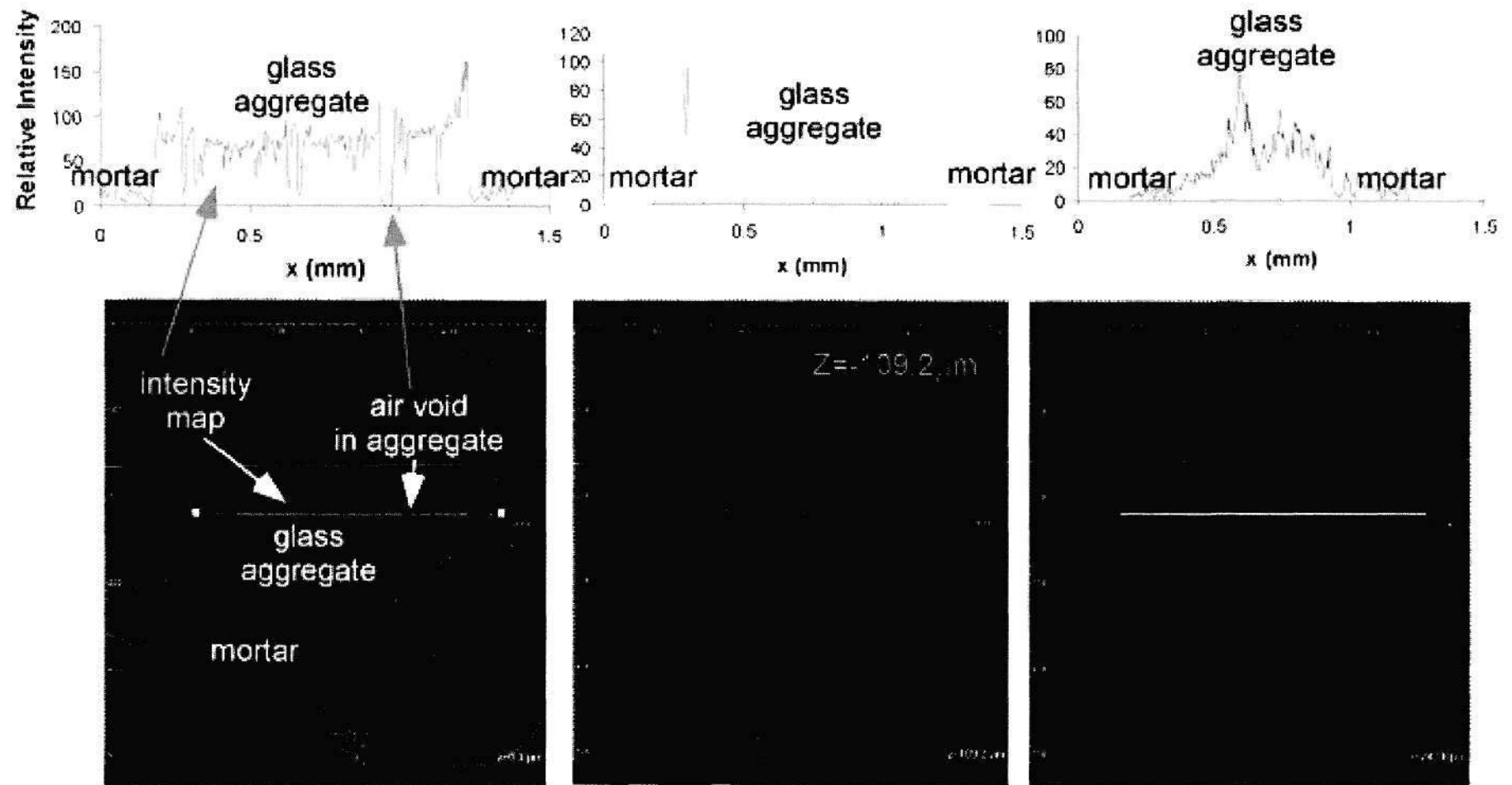


(4) rotation

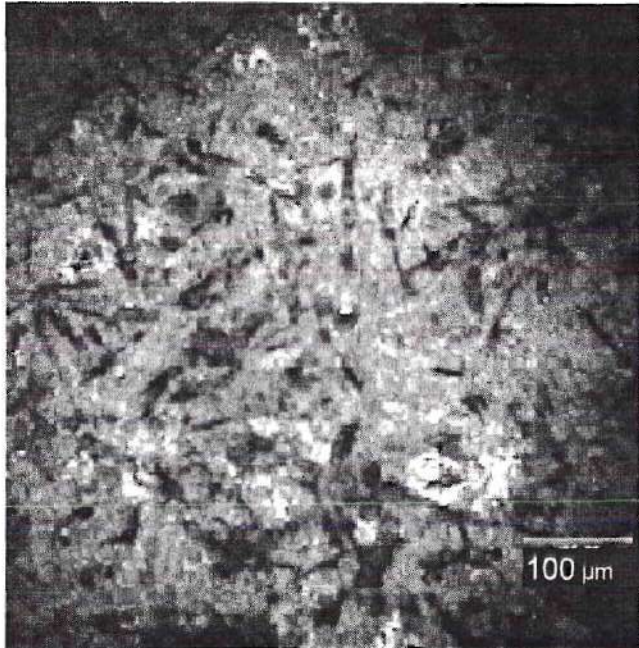


(5) y-z plane

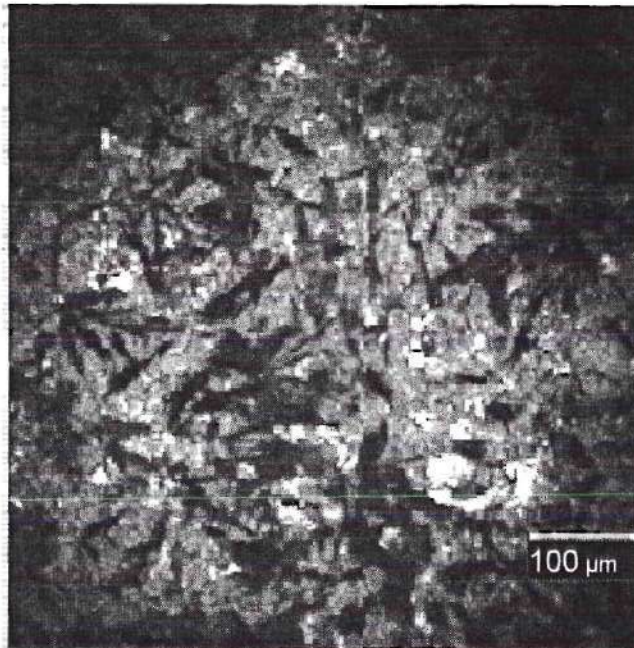
Through-Aggregate LSCM



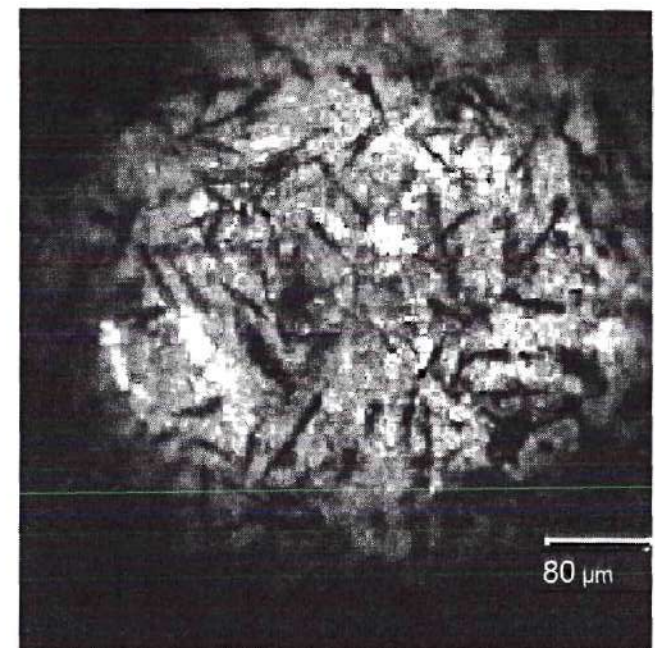
Through-Aggregate LSCM



LiNO_3 , $\text{Li}/\text{Na}=1.0$, 3 days



LiNO_3 , $\text{Li}/\text{Na}=1.0$, 8 days



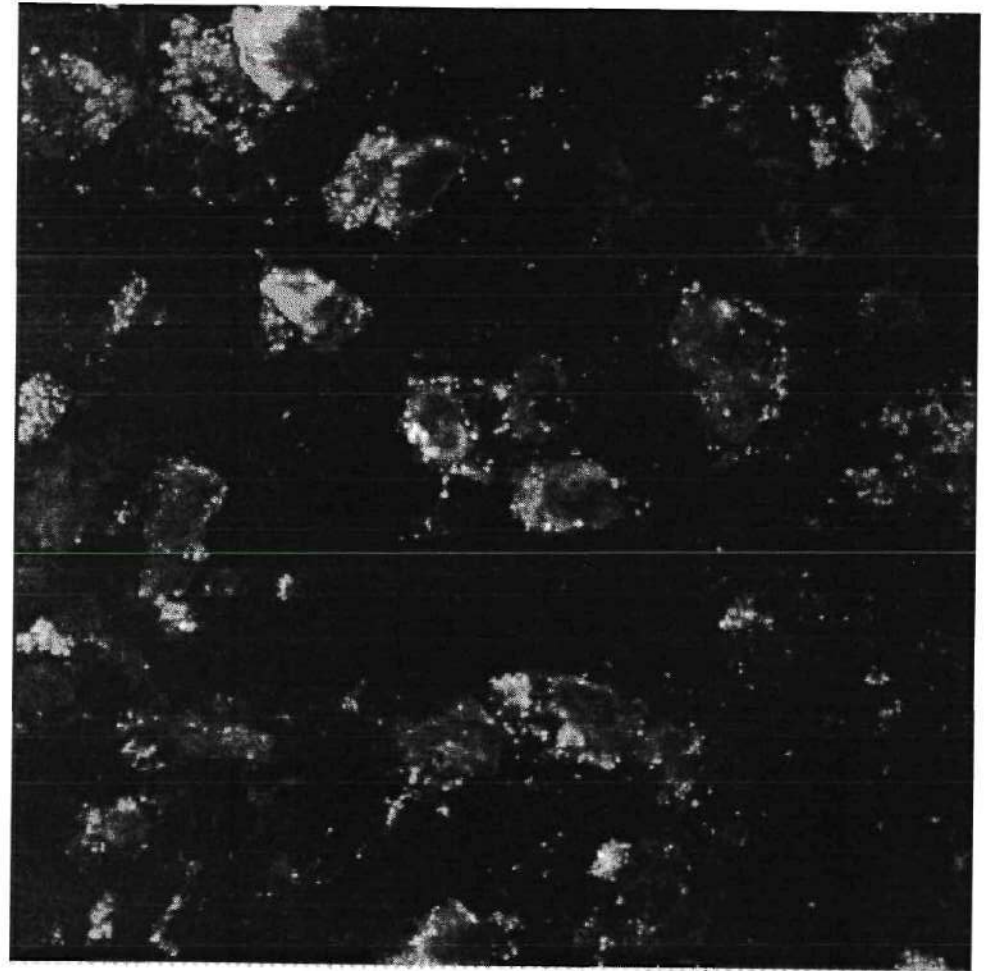
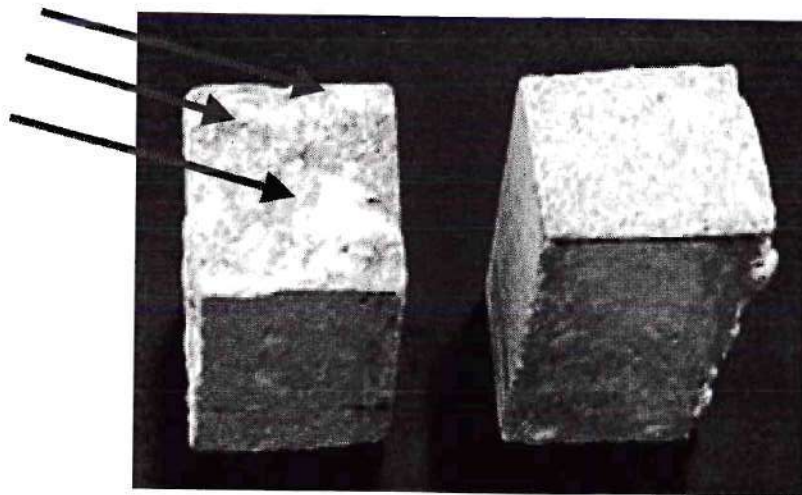
LiOH , $\text{Li}/\text{Na}=0.50$, 1 day

Lathlike reaction products apparent at the aggregate/cement interface in the presence of lithium only.

Fluorescence Imaging with Confocal Microscopy

Phase differentiation is one challenge with examination of composites.

Such data is necessary for volume fraction calculations, assessment of phase distribution, and characterization of interfacial properties, among others.



Summary

We examined the use of four techniques for characterization of cement-based materials:

Microtomography (μ CT) - lab based method used to examine small paste and mortar samples; may be used in combination with EDXRD

Transmission Soft X-ray microscopy (TXRM) - synchrotron method used primarily to examine dry powders and slurries through depth

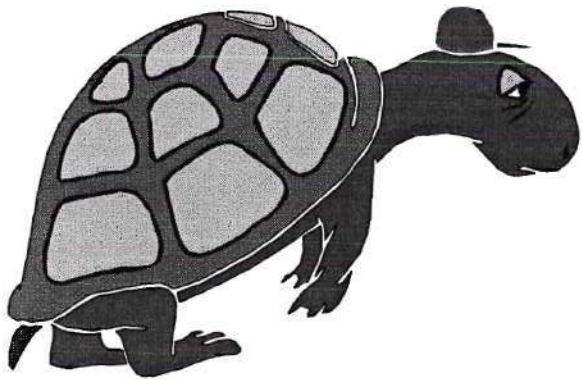
Laser scanning confocal microscopy (LSCM) - laser method used to examine a range of sample types, including fracture surfaces, polished samples, and slurries

Challenges and New Directions

- Three-dimensional characterization
- In situ, non-destructive, sub-surface evaluation of larger samples at high resolution
- Nanoscopy
- Techniques which allow for *in situ* process monitoring
- Multiple sensors
- Virtual microscopy
- Many, many, others...

Thank you!

"The best way to have a good idea is to have lots of ideas."
- Linus Pauling



1.0 Research resulting from confocal microscopy accessories acquired under MRI award

Three-dimensional models of material microstructure can be generated using confocal microscopy. The development of this technique has been underway to study several sample types, related to microstructure and durability concrete and other cement-based materials. In these types of samples, of particular interest are interfaces (e.g., aggregate/hydrated cement paste matrix in concrete), rough surfaces (e.g., fracture surfaces), and wet chemistry samples (e.g., cement hydration, pozzolanic reaction). An overview of the use of LSCM to examine each of these types of samples is given in [Kurtis *et al.* [2002]. Below, through-aggregate imaging is a technique which allows imaging of interfaces and characterization of fracture surfaces by LSCM are described.

Through-aggregate imaging

A new technique has been developed to allow imaging through glass aggregate in mortar samples by laser scanning confocal microscopy (LSCM). We have used through-aggregate imaging by LSCM to monitor in situ the evolution of damage in mortar samples from alkali-silica reaction. The re-examination of site over time with precision was allowed by the purchase of an indexable stage under MRI Award CMS-0079589. Also purchased under this award, the imaging software Leica 3D was used to quantify damage in these samples over time. Through-aggregate imaging of polished mortar samples containing glass aggregate and various amounts of lithium additives was performed with a Laser Scanning Confocal Microscope (LSCM). Sample preparation protocols and characterization methods are described herein, and have been described in greater detail in [Collins *et al.*, in press].

Sample Preparation

Mortar samples were prepared in accordance with ASTM C 227 except that the specified graded aggregate was replaced with borosilicate glass beads (similar to ASTM C 441), and sodium hydroxide was added to the mix water to increase alkali content (based on ASTM C 1293). The mortars were prepared with Type I cement, a water-to-cement ratio (w/c) of 0.37, borosilicate glass beads, and various quantities of lithium additives. NaOH was added to the mix water to increase the $\text{Na}_2\text{O}_{\text{equivalent}}$ to 1.0 wt. %. Graded round borosilicate glass beads (obtained through Fisher Scientific), with diameters of 1, 2, and 3 mm, were used in place of traditional aggregate to allow for through aggregate imaging. In addition to a control prepared without lithium additive, samples were prepared with a specific lithium additive (LiOH, LiNO_3 , or LiCl) added by molar concentration to the mix water, so the effect of each respective additive could be compared. The lithium additive dosages tested were $[\text{Li}_2\text{O}]/[\text{Na}_2\text{O}_{\text{eq}}] = 0, 0.5, \text{ and } 1.5$ (LiNO_3 only). The mortars were cast in 1x1x11.25in (25x25x285mm) mortar bar prism molds, which were then cut to approximately 1.25 to 1.5 in (31.75 to 38.1 mm) in length prior to polishing.

Polishing Method

After curing for 24 ± 2 hours in a humid container at $23.0 \pm 2.0^\circ\text{C}$, the samples were saw-cut axially using a water-cooled diamond saw. The cut surfaces were polished using sand paper grit sizes 120, 240, 320, and 600 and water as the lubricant, on a Buehler ECOMET 4 metallography grinder/polisher. It should be noted that the sample was not impregnated with epoxy prior to polishing, as is traditionally done, in order to allow the alkali-silica reaction to continue. Due to imperfections in the glass aggregate surface generated during the initial polishing step, a method to use CeO_2 powder (2 μm particles, obtained from His Glassworks, Inc.) mixed with water to a paste consistency (volume ratio of about 0.75 powder / 0.25 water), with a felt bob drill attachment to further polish the glass surface was developed. The cylindrical bob, 1.5 inches in length by 1.5 inches in diameter, was made of compact felt and was obtained from Duro-Felt Products, Inc. The flat end of the bob was used for polishing. After about five minutes of polishing, the sample was rinsed thoroughly, patted dry, and the surface of the glass was inspected for imperfections under the microscope. If necessary, the process of polishing with cerium oxide was repeated until scratches in the glass surface were largely eliminated. This allowed light from the microscope to penetrate the surface of the glass beads, and not refract due to imperfections. After this final glass polishing, images using the LSCM could be obtained through the entire depth of the aggregate to the paste-aggregate interface below the aggregate. In order to prevent interference with the time-dependent reaction products,

no further polishing was done after the initial polishing treatment. Polished mortar samples were mounted, with heat and moisture-resistant epoxy (Sikadur 32 High-Mod) to aluminum templates drilled with three holes. The three holes were spaced so that the template could fit on a custom-made sample stage with corresponding raised pegs in a unique position. This allowed positions to be revisited over time using an x-y indexable sample stage and coordinate reader from Boeckeler Instruments. Mortars were stored in humid metal containers, RH \approx 90%, at 38.0 ± 2.0 °C and were monitored over time (per ASTM C 227).

LSCM Method

LSCM images were obtained with a Leica TCS NT microscope powered by a single Ar laser ($\lambda = 488$ nm) in reflected light mode. A schematic representation of LSCM is depicted in Figure 1. First, the eyepiece was focused on the surface of the sample. Then the microscope was turned to scanning mode and the photo multiplier tube (PMT1) detector gain value, which controls the detector voltage, was adjusted such that there was no, or very little, image saturation. Image saturation was determined using the glow overf color look up table (LUT), where over-saturated pixels appear bright blue. The PMT1 value was only determined in this manner for the images obtained at 1 day at each x-y location. Consecutive images were obtained over time using the same PMT1 value that was used initially at each location. Images were taken with 2.5x/0.07 N PLAN, 5x/0.15 HC PL FLUOTAR, and 20x/0.40 H PLAN objective lenses. The imaging locations were mapped in the x and y planes using a cross hair, indexable stage, and coordinate reader. Images of each sample at each distinct x-y location were obtained at approximately 1, 7, 14, 28, and 56 days. Images at each x-y location were taken through successive z-depths of the glass aggregate. Three-dimensional (3D) images and relative reflected light intensity maps were generated using Leica Confocal Software Version 2.0.

Results

LSCM proved to be a useful method for monitoring reactions in mortars prepared without and with lithium additive. The control sample was prepared without the addition of a lithium additive to determine the base case of damage due to ASR gel expansion so the effects of lithium additives could be assessed. Damage commonly associated with ASR, such as gel rings around the aggregate, aggregate debonding, and map cracking through the paste and aggregate, was observed only in the control mortar sample. Over time, cracks grew in number and in width due to continuing ASR damage in the control sample. Figure 1 shows LSCM images, using a 20x/0.40 H PLAN objective, of cracking in the aggregate and paste progressing over time. Figure 5a shows the aggregate, undamaged, at 2 days. Figures 1b, c, and d show the gradual cracking and crack widening in the aggregate and the paste. Crack widening in the paste alone was also observed by LSCM.

Reactions in mortar samples prepared with lithium additives were also monitored over time by LSCM. Over the period of examination, samples prepared with lithium showed no cracking but a change in the paste/aggregate interface was observed, as is seen in Figures 2a-c obtained with a 20x/0.40 H PLAN objective at 3, 8, and 14 days. It is believed that the change observed at the paste/aggregate interface demonstrates the formation of a chemical reaction product, rather than being an indication of aggregate debonding. Debonding of the aggregate from the paste in the control sample resulted in a mean LSCM relative intensity less than 1, and here the mean intensity varies between approximately 12 and 50. The products at the interface appear to have ordered structures and possess lathlike or dendritic morphologies. Based on these observations, it is proposed that they may be crystalline in nature. The amount of these products formed at the interface typically diminished over time. Each individual dendrite appears to be less than 80 μ m in length. These products were first noticed at 1 day of age after demoulding and polishing and were present in all samples prepared with Li, independent of salt type. However, the quantity of the product and how it changed with time, varied with location and lithium additive type. LSCM images of the paste/aggregate interface of samples prepared with LiNO_3 , $[\text{Li}_2\text{O}]/[\text{Na}_2\text{O}_{\text{eq}}] = 0.5$, taken over 14 days show the reaction product growing in size.

Remarks on Efficacy of New Technique

The use of through aggregate imaging by laser scanning confocal microscopy (LSCM), a technique for imaging reactions in concrete through glass aggregate, was developed and was shown to be effective for examining alkali-silica reaction in situ. Three-dimensional representations of the aggregate, images of reaction product both within cracks and at the paste/aggregate interface, and quantitative

measurement of gel ring thickness at the surface are all examples of types of information gained by LSCM that have not, at this point in time, been possible with other microscopy methods.

LSCM images showed definitive evidence that ASR occurred in the sample prepared without lithium additive, characteristic crack patterns, gel rings, common reaction products, and debonding were observed. Samples prepared with lithium showed no evidence of ASR. However, through aggregate imaging showed reaction product formation occurring at the paste/aggregate interface in which apparently crystalline reaction products were formed, and it is likely that these products play a key role in mitigating expansion due to ASR in the presence of lithium.

Fracture Surface Characterization

Much of the existing research using confocal microscopy to examine cement-based materials has focused on surface characterization. For example, Lange and co-workers have explored the correlation between fracture surface roughness and fracture energy. A roughness number (RN) is a quantification of the roughness of a surface; RN can be generated from surface topographies acquired by confocal microscopy. RN is the area of a triangulated surface (A_t) compared to the corresponding nominal area (A_p): RN is the area of a triangulated surface (A_t) compared to the corresponding nominal area (A_p):

$$RN = \frac{\sum A_t}{\sum A_p}$$

It follows, then, that a planar surface has RN equal to one. Also, RN will generally increase as surface scanning intervals or resolution decrease.

We have recently started using confocal microscopy to measure surface roughness of fracture surfaces resulting from flexural testing of fiber-cement beams. Our goal is to examine whether a correlation may exist between RN in these samples and their measured toughness. Samples have been prepared with a consistent fiber volume fraction and water-to-cementitious materials ratio, but with varying amounts and varying types of supplementary cementitious materials, including Class C and F fly ash, slag, and silica fume. Some preliminary results, shown in Figure 3, suggest that correlations likely exist between RN and toughness for these types of samples, although some anomalies are found in the data for 50% Class C fly ash samples.

Additional work is being done to assess the effects of wet-dry cycling on fiber-cement mechanical properties. Here, RN is being tracked in samples tested at the same age (to avoid changes in matrix characteristics with time) but after varying numbers of wet/dry cycles. Data in Figure 4 shows that RN, like toughness, does decrease with cycling, particularly beyond 2-3 cycles.

2.0 Research Resulting from the Acquisition of IR Imaging System and Photoelastic Imaging Device

DT1400 Infra-Red Thermal Imaging System (256 x 256, Liquid Nitrogen cooled)

System Description:

This system consists of a very sensitive infrared camera that is combined with special high-speed digital electronics to measure small changes in a temperature field. The result is a full-field stress map of the surface. This system includes a motion compensation software and hardware; it allows for attained compensation for motions at frequencies from 0.6-400Hz. The system is also capable of following a variable amplitude (random) motion. The detector array contains thousands of on-chip integrators that collect data simultaneously, producing a near-live full-field stress image. Data collected from the infrared camera head is processed at hundreds of frames per second. The processed images can be used to measure properties that are directly related to the principal stress field and/or to temperature profile.

The DT 1400 camera head is based on an InSb focal plane array. The array is cooled to liquid nitrogen temperature. The system provides excellent DC thermal imaging, and very high quality AC performance. System includes camera head, cables, computer, and image processing boards. Stress Photonics DV Foundation image processing software, DeltaTherm Control Panel, and DeltaVision post-acquisition

analysis data-processing software is included: Also included are tripod and carrying cases for computer and DeltaTherm.

Features of the system (in addition to those stated above):

- Live, high quality DC thermal imaging with corrections for detector uniformity to make setting up microscopic and other imaging a simple matter of point, focus, and shoot.
- Built in oscilloscope for checking the quality of the reference signal.
- Thermal Resolution: 1mKelvin full-field (30s acquisition time)
- Constant amplitude and variable amplitude load sequences with impact triggering
- Loading frequencies 0.6 Hz to 400 Hz
- Capable of spatial resolutions better than 23 micron per pixel
- DeltaVision software for data acquisition, presentation and storage.
- 12 month system warranty and service contract

GP 1200 Photoelastic Stress Analysis System

System Description:

The GFP1200 Photoelastic stress/strain analysis (PSSA) System is a strain measurement system based on photoelasticity. Advanced instrumentation is used which differentiates this system from similar photoelastic techniques used historically. A specially tinted paint-on coating is used for this purpose. A new technique is developed for measuring the thickness of a paint-on coating automatically. The same automatic polariscope that measures the strain amplitude can measure the thickness of the coating, eliminating it as an unknown. After a thin plastic coating is applied to the surface of the part, an instrument called a polariscope measures the strain-induced birefringence to create a full-field strain map. A software is been developed with the system in order to allow data interpretation and post analysis.

The system includes:

- Enhanced Grey-Field Polariscope with automated filters.
- Polarized light projector.
- Windows NT computer with LCD monitor.
- Enhanced DeltaVision software for post-acquisition analysis and presentation of images.
- GFP virtual control panel for image acquisition.
- Matrox Image Acquisition Card.
- Tripod and tripod head.
- Coating Kit.

Partially helped Acquire: MTS Model 810 Material Testing System

This system is a uniaxial hydraulic loading frame used to load structural components and material coupon samples while the GFP-1200 and Delta Therm 1400 are monitoring the testing sample. The main features of this hydraulic loading frame are:

- 1) Nominal dynamic load rating: 250 kN (55,000 lbs).
- 2) 55kip Actuator, 6" Stroke
- 3) Hydraulic grips with adjustable pressure
- 4) 4" and 2" wedge sets
- 5) 6-channel input and output for data acquisition
- 6) software for programing tests
- 7) Low noise "silent flow" hydraulics
- 8) Special output card for interfacing with the Delta Therm 1400 and GFP1200 systems
- 9) MPT software and control